

DOE/CBFO-01-1005

# **Performance Demonstration Program Plan for Nondestructive Assay of Drummed Wastes for the TRU Waste Characterization Program**

**Revision 1  
August 2005**



**U.S. Department of Energy  
Carlsbad Field Office  
Office of Characterization and Transportation**

**This document supersedes Revision 0.1 of  
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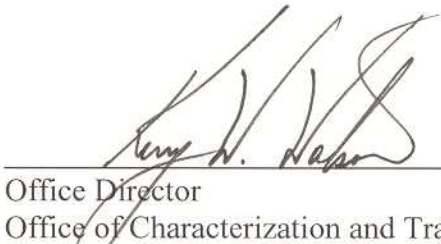
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August 2005**

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## ACRONYMS AND ABBREVIATIONS

%R	percent recovery
%RSD	percent relative standard deviation
CBFO	Carlsbad Field Office
DOE	U.S. Department of Energy
FGE	fissile gram equivalent
MDC	minimum detectable concentration
NDA	nondestructive assay
OCT	Office of Characterization and Transportation
PDP	Performance Demonstration Program
QA	quality assurance
QAO	quality assurance objective
QAPD	<i>Quality Assurance Program Document</i>
SNM	special nuclear material
SOP	standard operating procedure
SPT	sample preparation team
TID	tamper indicating device
TRU	transuranic
TWBIR	<i>Transuranic Waste Baseline Inventory Report</i>
WAC	<i>Waste Acceptance Criteria for the Waste Isolation Pilot Plant</i>
WIPP	Waste Isolation Pilot Plant

## **Significant Changes to the Drum Waste NDA PDP Plan, Revision 2**

### **General**

This document supersedes Revision 0.1 of the *Performance Demonstration Program Plan for Nondestructive Assay for the TRU Waste Characterization Program* (DOE/CBFO-01-1005), and is a complete rewrite of that document. The document was rewritten to:

1. Eliminate the criteria for the identical assay systems (Appendix A)
2. Eliminate the requirement for a sample disassembly form.
3. Remove the requirement to use the electronic data recorder.
4. Reflect recent changes in the Carlsbad Field Office (CBFO) organizational chart.

This revision constitutes a complete revision of each page, therefore no change bars are included.

## 1.0 INTRODUCTION

### 1.1 General

The Performance Demonstration Program (PDP) for Nondestructive Assay (NDA) is a test program designed to yield data on measurement system capability to characterize drummed transuranic (TRU) waste generated throughout the Department of Energy (DOE) complex. The tests are conducted periodically and provide a mechanism for the independent and objective assessment of NDA system performance and capability relative to the radiological characterization objectives and criteria of the Office of Characterization and Transportation (OCT). The primary documents requiring an NDA PDP are the *Waste Acceptance Criteria for the Waste Isolation Pilot Plant* (WAC), which requires annual characterization facility participation in the PDP, and the *Quality Assurance Program Document* (QAPD). This NDA PDP implements the general requirements of the QAPD and applicable requirements of the WAC.

Measurement facilities must demonstrate acceptable radiological characterization performance through measurement of test samples comprised of pre-specified PDP matrix drum/radioactive source configurations. Measurement facilities are required to analyze the NDA PDP drum samples using the same procedures approved and implemented for routine operational waste characterization activities. The test samples provide an independent means to assess NDA measurement system performance and compliance per criteria delineated in the NDA PDP Plan. General inter-comparison of NDA measurement system performance among DOE measurement facilities and commercial NDA services can also be evaluated using measurement results on similar NDA PDP test samples.

A PDP test sample consists of a 55-gallon matrix drum containing a waste matrix type representative of a particular category of the DOE waste inventory and nuclear material standards of known radionuclide and isotopic composition typical of DOE radioactive material. The PDP sample components are made available to participating measurement facilities as designated by the Carlsbad Field Office (CBFO).

The nuclear material type, mass and associated alpha activity of the NDA PDP radioactive standard sets have been specified and fabricated to allow assembly of PDP samples that simulate TRU alpha activity concentrations, radionuclidic/isotopic distributions and physical forms typical of the DOE TRU waste inventory. The PDP matrix drum waste matrix types were derived from an evaluation of information contained in the *Transuranic Waste Baseline Inventory Report* (TWBIR) to ensure representation of prevalent waste types and their associated matrix characteristics in NDA PDP testing.

NDA drum analyses required by the Waste Isolation Pilot Plant (WIPP) may only be performed by measurement facilities that comply with the performance criteria as set forth in the NDA PDP Plan. In this document, these analyses are referred to as WIPP analyses, and the wastes on which they are performed are referred to as WIPP wastes.

### 1.2 Purpose

The purpose of the NDA PDP for drummed waste is to provide independent data on the ability of NDA measurement facilities (including DOE owned or contracted systems) to meet the data quality objectives for NDA of TRU wastes intended for disposal at WIPP. The CBFO uses the drum NDA PDP as part of the assessment and approval process for measurement facilities characterizing waste for the OCT. In addition to the NDA PDP, this process includes technical evaluation of NDA measurement system implementation and use, method and performance data supplied by the measurement facility, and independent technical quality assurance audits.



This NDA PDP Plan describes the elements comprising the test program, including test apparatus, test sample configuration, and required sample analyses. This NDA PDP Plan also identifies criteria used to evaluate NDA measurement system performance and the responsibilities of the program participants, including the NDA PDP coordinator, sample preparation team (SPT), measurement facility contact responsibilities, and the measurement facility as an entity. The CBFO implements the plan by designating an NDA PDP program coordinator and by providing program oversight and coordination.

### 1.3 Scope and Frequency

An annual NDA PDP test cycle, called a primary cycle, is administered to all facilities actively engaged in radiological characterization of WIPP wastes for the OCT. Facilities actively engaged in radiological characterization of WIPP wastes must participate in the primary cycle. Test samples for the primary cycle are specified by the NDA PDP coordinator for each participating measurement facility. For measurement facilities unable to participate at the time the primary cycle is conducted (for reasons such as facility operational conflicts or system modification) supplemental cycles may be provided. A measurement facility unable to participate in the primary cycle must submit a request to the CBFO for either a time extension to participate in the primary cycle, or for a supplemental cycle. Should an extension be granted, test results from the requesting facility will be included in the primary PDP cycle report. If the CBFO grants a supplemental cycle, a separate test is conducted and a separate report prepared. The cost of the supplemental cycle is typically borne by the requesting facility. Supplemental cycles may also be conducted any time at the discretion of the CBFO, including at measurement facilities that have successfully completed the primary cycle for that year.

Unless a specific exemption is granted by CBFO, NDA measurement facility participation in the NDA PDP is required in accordance with the following:

- All NDA systems in use at the time of a scheduled primary cycle must participate in that cycle or request an extension.
- Any NDA system unable to participate in the primary cycle must request a supplemental cycle.
- Every measurement system must go through an initial NDA PDP cycle in order to establish confidence that the system is acceptable to characterize TRU wastes for the OCT. An “initial PDP cycle” is defined as, but not limited to: 1) the first implementation of an NDA measurement system; 2) initial use after significant modification or recalibration; 3) application of an NDA system to waste forms significantly different than those for which it is certified; and 4) movement to another location.
- NDA systems that have successfully participated in the current year primary cycle and are relocated to a site where no PDP standards and drum set exist, will be allowed to perform WIPP waste radiological characterizations if the CBFO verifies and approves the system. Documentation on system capability and performance relative to the waste forms at the new site must be submitted to CBFO for review and approval. The system must have successfully participated in an NDA PDP cycle within the last 13 months.
- NDA PDP quality assurance objectives, in addition to other criteria deemed appropriate by the CBFO, will be used to determine initial acceptability of measurement system function and performance prior to use for WIPP waste characterization. During the initial cycle, the measurement system may be subject to more PDP test samples than typically employed in a primary cycle.
- Inactive NDA systems are defined as certified systems not being used to characterize WIPP wastes. Inactive systems are not required to participate in NDA PDP cycles.

- All systems classified as inactive during a primary PDP cycle must participate in a NDA PDP cycle prior to return to active status. If the reactivation schedule does not coincide with the next scheduled primary PDP cycle, the owner/operator must petition the CBFO for a supplemental cycle.

The PDP samples must be analyzed using methods the measurement facility has implemented or plans to implement for routine analysis of WIPP wastes. Only the methods actually used in the PDP are considered acceptable to support the analysis of WIPP wastes.

## 1.4 Radioisotopes, Activities, and Waste Matrices

The following primary isotopes are evaluated under the NDA PDP Plan. Of these, the first four are the most significant in terms of WIPP inventory parameters and limits, and are subject to scoring per this plan.

- $^{238}\text{Pu}$
- $^{239}\text{Pu}$
- $^{240}\text{Pu}$
- $^{241}\text{Am}$
- $^{233}\text{U}$
- $^{234}\text{U}$
- $^{238}\text{U}$

The uranium isotopes comprise a significant fraction of the DOE inventory and must be accounted for in the repository assessments, but are not subject to scoring. Therefore, PDP oxide powder standards containing plutonium (powder and granular forms), americium, and depleted and enriched uranium have been specified, fabricated, and distributed to select sites to support NDA PDP program implementation.

The 55-gallon drums used with the PDP standards to create test samples contain matrix materials representative of DOE TRU waste types. These matrix materials are configured and fixed within the PDP matrix drum, in a manner replicating the nominal properties of an actual waste matrix type in terms of material composition, density, and density distribution. In this manner the drum matrix surrogates replicate the physical properties of real waste forms and manifest perturbations in the response of NDA systems, as do actual wastes.

The TRU waste forms present in the DOE inventory comprise a broad spectrum of waste types. To represent this range of waste types, several different surrogate waste matrix drums were specified and fabricated. Specification of the PDP matrix drum waste types was derived from the 11 waste form categories defined in the TWBIR. The material properties of the 11 TWBIR waste categories were considered in terms of their respective effects on NDA technologies and were subsequently condensed to five matrix types: noninterfering matrix (empty), combustibles, mixed metals, glass, and solidified inorganic sludge.

## 2.0 PROGRAM COORDINATION

### 2.1 General Responsibilities

The CBFO is the review and approval authority for the NDA PDP for drummed DOE waste. Programmatic direction and oversight of the PDP are performed by the OCT, which manages the PDP on behalf of the CBFO. The OCT is part of the Office of Operations. Figure 1 summarizes the organizational flow of the NDA PDP program.

A CBFO-designated organization functions as the program coordinator and technical advisor to the CBFO. The program coordinator must comply with the responsibilities stipulated in this plan in accordance with the requirements of the CBFO *Performance Demonstration Program Management Plan*. The NDA PDP coordinator is responsible for the following:

- Distributing and tracking PDP standards and matrix drums as appropriate to implement the program.
- Maintaining documentation on all PDP standards and matrix drums.
- Confirming the schedule of a primary PDP cycle at least 2 weeks before the planned start date.
- Distributing PDP sample preparation instructions and associated materials.
- Ensuring training for the on-site SPTs is in accordance with the NDA PDP Plan.
- Collecting and scoring PDP sample data reports from the measurement facilities.
- Preparing cycle-specific measurement system scoring reports.
- Ensuring that records of participation and results of all PDP cycles are adequately maintained in a retrievable condition.
- Reviewing changes in the QAPD or WAC that affect this plan and revising as appropriate, per CBFO direction.
- Assisting in the resolution of disputes related to NDA system measurement results, test sample configuration, operational restrictions, and other issues that may arise during a PDP test cycle.
- Developing technical recommendations to the CBFO regarding conditional NDA characterization approvals related to PDP performance.
- Maintaining a current list of participating facilities and their measurement system registrations. The registration system contains information on each assay system including a unique identity, measurement principles and modes, the PDP test combinations permitted under facility/system-specific calibration, and administrative limits.
- Preparing a periodic NDA PDP performance update document that contains a compilation of results of every NDA PDP cycle conducted since inception of the program through to the end of the current calendar year. The document includes all participating measurement facilities, measurement systems, and test samples for all cycles, including supplementals. Recorded data, pertinent trending plots regarding performance parameters, and test sample configuration are to be addressed.
- Ensuring the NDA PDP test material inventory at each site is sufficient to support cycle implementation.
- Procuring NDA PDP components to replace lost or damaged parts as necessary.

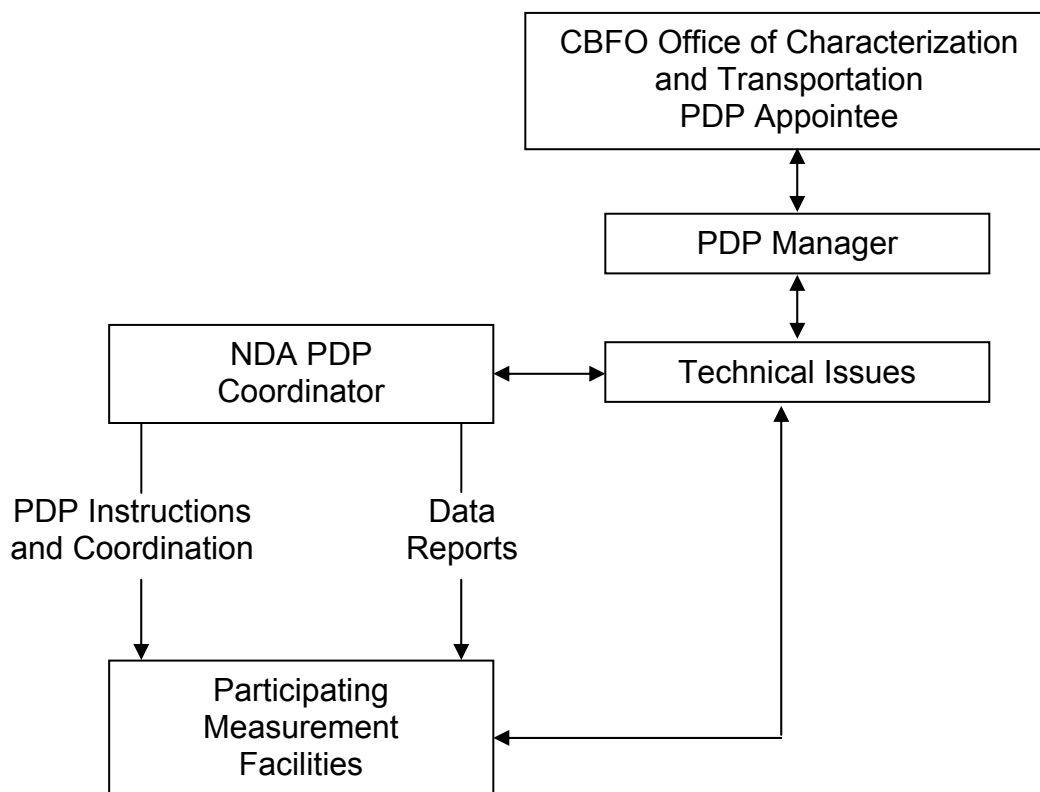


Figure 1. Organization and information flow for the NDA PDP.

The CBFO grants approval for each measurement facility/system participating in the NDA PDP. Facilities/systems that are not current participants may petition the CBFO to participate in the PDP. Participation by measurement facilities not actively engaged in characterization of TRU wastes for WIPP-related programs is at the discretion of the CBFO.

### 2.1.1 Measurement Facility Contacts and General Responsibilities

Each participating facility is required to provide the NDA PDP coordinator, in writing or by e-mail, with the name, telephone number, fax number, and address of a single contact person (assay coordinator) responsible for on-site administration and conduct of the NDA PDP (e.g., cycle-specific schedules and instructions). The assay coordinator is to be a full time employee or subcontractor of the characterization facility. The assay coordinator is responsible for ensuring necessary communication, verbal and/or written, with the NDA PDP coordinator regarding all aspects of the conduct of a given PDP cycle.

The measurement facility is also required to provide a contact and address suitable for freight and package delivery of the matrix drums, PDP standards, and renewable parts. Such information shall be supplied in writing or e-mail, each time the contact personnel change.

Each participating facility is required to provide the NDA PDP coordinator, in writing or by e-mail, the inventory and location of all PDP materials (standards, drums and parts) twice a year (January and July). A description of the condition of the PDP sample components is also required (e.g., TID security lanyard and locating pin condition). Digital images of the PDP sample components can be submitted in lieu of condition descriptions. Any changes in personnel should be noted along with the inventory.

Participants must complete a registration form for each measurement system to be tested in the NDA PDP to establish its identification, calibration range, operational limits, and other administrative information affecting NDA PDP testing. Measurement systems must be registered at each measurement facility (some DOE sites have multiple measurement facilities) such that the registration is unique to the application of that system at a specific measurement facility. Appendix A contains the necessary forms and instructions for registering a system for the NDA PDP.

## **2.2 Program Assessment**

The NDA PDP is routinely assessed for utility and effectiveness through several interrelated activities. These activities include the OCT's review of the test results for each NDA PDP cycle; approval of this plan; consideration of the reports and observations of the program coordinator, PDP manager, and NDA PDP coordinator; feedback from program participants; and comments from other parties such as independent quality assurance (QA) assessors. Records of comments, decisions, or proposed actions resulting from the OCT assessment may take any documented form, including, but not limited to, routine program correspondence, meeting minutes, action items, formal review of program documents, assessment reports, and corrective actions.

## **2.3 Procurement**

Procurement activities necessary for conducting the NDA PDP must comply with the QAPD. In accordance with the QAPD, the responsible purchasing organization maintains all procurement documents and performs all procurement activities.

## **2.4 Training**

Each organization involved in the implementation of the PDP shall meet the training requirements of the QAPD. Organizations shall retain on file evidence that: 1) personnel have the necessary program documents (controlled or uncontrolled, as applicable) for their use; and 2) personnel have read and understand program-governing documents pertinent to their duties in support of the NDA PDP. At a minimum, these documents include applicable portions of the QAPD, the WAC, and this plan.

### 3.0 PREPARATION AND DISTRIBUTION OF RADIOACTIVE STANDARDS AND MATRIX DRUMS

#### 3.1 Overview

The CBFO is responsible for the specification and procurement of PDP standards. A PDP standard is defined as a radioactive source designed, prepared (or acquired), and certified for the NDA PDP. PDP standards have pedigrees traceable to nationally recognized reference bases such as the National Institute of Standards and Technology. To date, all standards used in the NDA PDP have been manufactured at the Los Alamos National Laboratory. However, sources with appropriate traceability and certification from other programs or commercial sources may be used. The types of radioactive PDP standards presently in the NDA PDP inventory are listed in Table 1.

The CBFO is also responsible for the specification and procurement of PDP matrix drums for use in the NDA PDP. A PDP matrix drum is a standard 208-liter (55-gallon) waste drum containing matrix materials in a configuration representative of DOE-generated wastes.

NDA PDP standards, matrix drums, and matrix drum components are not to be used for any activity other than conduct of the PDP cycle, as specified by the NDA PDP coordinator. Use of these components for any other purpose must have written approval from the CBFO.

#### 3.2 PDP Radioactive Material Standards

The external dimensions of the PDP standard are compatible with PDP matrix drum insert fixtures that provide for the secure and accurate placement of standards within the 55-gallon matrix drum. Refer to Appendix B for general specifications of the PDP radioactive standards currently inventoried. Detailed information and data concerning the specification, design, fabrication, and traceability for each PDP standard manufactured in each production phase is provided in their respective production plan documents (see references). As indicated in Table 1, there were seven different PDP standard production campaigns, each producing PDP standards with differing nuclear material content.

Table 1. NDA PDP radioactive standard inventory.

Type of Activity		Production Phase
1.	Weapons-grade plutonium (low mass set)	Phase I
2.	Weapons-grade plutonium (high mass set)	Phase II.A
3.	Large particle size weapons-grade plutonium	Phase II.B
4.	Enriched uranium	Phase II.C
5.	Depleted uranium	Phase II.D
6.	Increased Am/Pu ratio (relative to the native quantity in weapons-grade plutonium)	Phase III.A
7.	Heat source plutonium	Phase III.B

### 3.3 PDP Surrogate Waste Matrix Drums

The NDA PDP matrix drums are identified as to matrix type and serial number for the PDP. One of the PDP drums is a zero (empty) matrix drum containing only the fixtures necessary to reliably and reproducibly position the PDP standards and a 90 mil polyethylene liner. The remaining matrix drums contain surrogate matrix materials replicating that range of waste matrix properties listed in the TWBIR that affect NDA measurement system response. The NDA PDP matrix drum inventory consists of the following:

- Noninterfering matrix (empty) drum, serial# 001
- Combustible waste, serial# 003
- Glass waste, serial# 004
- Solidified inorganic sludge, serial# 005
- Mixed metals waste, serial# 006

Refer to Appendix C for general information on the PDP matrix drum design and configuration. Detailed information regarding the specification, design, and as-built data for the NDA PDP matrix drums is provided in the *Design of Interfering Matrix Drums for the Non-Destructive Assay Performance Demonstration Program for the National TRU Program*, INEEL/EXT-02-00320, Idaho National Engineering and Environmental Laboratory, Idaho Falls, Idaho, February 2002.

### 3.4 PDP Sample Component Control and Use

The NDA PDP coordinator shall ensure delivery of the appropriate set of PDP standards and matrix drums to each measurement facility prior to that facility's participation in PDP measurement activities. The NDA PDP coordinator shall ensure that any lost or damaged PDP components are replaced to prevent delay of a PDP cycle. The measurement facility is responsible for assigning a secure storage area for all PDP components and for meeting applicable safeguards, security, and safety requirements. No one is authorized to remove the PDP matrix drum lids and tamper with the matrix drum contents in any way without the express, written permission of the NDA PDP coordinator.

The PDP standards and matrix drums are not to be used for activity other than the performance of the NDA PDP. Requests for any other uses must be submitted to the CBFO in writing for each specific use requested. If a request is granted, CBFO will provide written approval. Under no circumstances are PDP standards to be used for measurement system calibration.

Before PDP standard shipment, each participant will make appropriate arrangements with the facility's safeguards and radiation safety organizations for storage and accountability. The SPT will coordinate with the site safeguards staff to comply with all site special nuclear material (SNM) requirements. At the time of receipt, the assay coordinator and/or SPT assigned by the measurement facility shall inspect, inventory, and secure the PDP standards, and will perform external inspections of the matrix drums and associated components for defects or damage.

The NDA PDP coordinator shall coordinate any transfers of sources between sites and no source movement will occur without written approval from CBFO. The NDA PDP coordinator will provide the transferring facility with the necessary contact information (names, phone numbers, e-mails, and physical addresses) for the receiving facility (e.g., another participating site). The current custodian will notify each site contact prior to the shipping date for PDP standards and/or matrix drums. The PDP standards will be sent to the address and individual designated by the receiving facility. The assay coordinator at

each site shall notify the NDA PDP coordinator in writing, with a copy to CBFO (e-mail is acceptable), of any changes in contact personnel.

### 3.4.1 PDP Sample Components Receipt

On receipt of PDP standards and/or matrix drums, the assay coordinator and/or the SPT shall:

- Verify that the serial numbers and physical descriptions of the PDP standards, matrix drums, and associated components received match those on the shipping manifest. PDP matrix drums are not to be opened. Inspection for damage is to be made only for removable components (e.g., insert fixture and drum exterior).
- Verify that the proper PDP components were received and not damaged. If a discrepancy in the shipping manifests or damage is found, the assay coordinator and/or the SPT shall secure the items and notify the NDA PDP coordinator. If no discrepancies or damage is found, the assay coordinator shall notify the NDA PDP coordinator in writing or by e-mail, that the shipment was received in acceptable condition.
- Once it is verified that the proper PDP components were received and not damaged. Ensure all NDA PDP components are properly stored and controlled to prevent unauthorized use, loss, or damage. Proper storage includes indoor, controlled climate (e.g., heated space), protection from exposure to water, and protection from physical damage (e.g., drum stacking, forklifts). Security is to be maintained on all PDP standards. PDP standards and PDP matrix drums are used only in accordance with CBFO policy. Questions concerning permissible use shall be referred to the CBFO or the NDA PDP coordinator.

### 3.4.2 PDP Sample Components Configuration Control and Repair

All PDP components must be maintained in the configuration in which they were provided to the participant. Any repairs to PDP drums or component parts must be coordinated with the NDA PDP coordinator. **Under no circumstances should any modifications be performed on the NDA PDP standards.** If minor repairs to drums and related materials are needed, the facility may make those repairs with NDA PDP coordinator approval. Replacement parts will be provided by the coordinator. Any significant repair will require that the drum or component be returned to the NDA PDP coordinator.



## **4.0 TEST SAMPLE PREPARATION**

### **4.1 Responsibilities**

A two-person SPT, consisting of a PDP standards custodian and a PDP standards configuration attestant, shall be assigned by each measurement facility. The assay coordinator may serve as a member of the SPT as long as independence from the measurement process is maintained. The SPT members must, at a minimum, possess the following qualifications and experience:

1. Full-time employee or subcontractor of the measurement facility.
2. Independent of the measurement group being tested; that is, neither member of the SPT or assay coordinator may participate in assay measurements of PDP samples that they have prepared.
3. QA trained, including applicable site QA training and the training provided through the NDA PDP program for SPTs.
4. Qualified to handle radioactive materials (PDP standards custodian only).

The PDP standards custodian and/or assay coordinator is responsible for coordinating on-site activities with safeguard organizations, radiation safety, and NDA PDP measurement facility contacts. These activities include, but are not limited to, PDP standard receipt, storage, and retrieval of PDP standards, control of stored components (e.g., PDP matrix drums, insert fixtures, and matrix spacers), PDP sample preparation, and PDP sample disassembly. During the conduct of a PDP cycle, the PDP standards custodian or assay coordinator notifies the NDA PDP coordinator should any issue develop regarding the use, control, and condition of the NDA PDP samples and ancillary items.

The PDP standards configuration attestant is responsible for verifying the proper placement of PDP standards within the matrix drum and security-related or other procedures associated with sample preparation. The PDP standards configuration attestant is responsible for ensuring that operations executed by the PDP standards custodian are performed in accordance with the standard preparation instructions supplied by the NDA PDP coordinator. To perform these functions, the PDP standards configuration attestant witnesses all PDP standard-loading operations and sealing of the loaded PDP sample drum using serialized, NDA PDP TIDs provided by the NDA PDP coordinator. The assay coordinator and/or SPT are also responsible for the integrity of the prepared PDP sample from the time it is sealed until disassembly at the conclusion of the cycle. Other than the SPT or assay coordinator, no observers are permitted during the PDP sample preparation process without the prior permission of the NDA PDP coordinator. The assay coordinator and/or SPT shall not divulge any information regarding the loading and configuration of the PDP sample until the test results are released by the CBFO or as indicated by the NDA PDP coordinator.

### **4.2 NDA PDP Sample Preparation Instructions**

At least two weeks prior to the scheduled start date of a primary PDP cycle, the NDA PDP coordinator shall forward a letter of instruction to the assay coordinator of each participating facility. This letter will specify the identification and position of each PDP standard to be inserted in each matrix drum to be used in the cycle. This information is supplied on the PDP sample configuration form for nondestructive assay (see Appendix D). For a primary NDA PDP cycle, the date the cycle commences is specified in the instructions provided to the assay coordinator. Measurement result report forms (see Appendix D) must be received within 28 days of the cycle start date.

When a supplemental cycle is conducted, the instructions will be sent by the NDA PDP coordinator to the facility assay coordinator. Within five working days of tracked receipt of the instruction package by the NDA PDP coordinator, the assay coordinator must check the package for completeness, and e-mail verification of receipt to the NDA PDP coordinator. The date this verification is received by the NDA PDP coordinator commences the measurement and reporting period for the supplemental NDA PDP cycle. Measurement result report forms must be received by the NDA PDP coordinator within 28 days of the cycle start date.

The following steps are repeated for the preparation of each PDP sample specified for a given cycle:

1. The SPT shall identify the correct PDP standards using the applicable sample configuration form. The sample configuration form provides the SPT with matrix-drum-specific PDP standard placement. The PDP standards configuration attestant shall verify that the proper standards were selected for PDP matrix drum configuration and that they were installed in the correct position.
2. The SPT shall select the proper serial-numbered 55-gallon matrix drum for installation of PDP standards and matrix spacers. The PDP standards configuration attestant shall verify that the proper matrix drum(s) were selected for PDP standard loading.
3. The SPT shall coordinate the placement of PDP matrix drums, PDP standards, and related equipment (such as TIDs) into a designated sample preparation area.
4. The SPT shall examine all required PDP sample components (e.g., matrix drums, PDP standards, matrix spacers) per the configuration form. The objective of the pre-load examination is to determine if any components are missing or damaged.

**NOTE:** If there is a damaged or missing PDP sample component, the SPT and/or assay coordinator shall take appropriate action as follows, depending on the component:

- Notify the NDA PDP coordinator of any PDP component loss or damage. If the component is a consumable item (such as a TID, form, insert tube, or matrix spacer), determine if a spare component is available from the site inventory. If so, replace the missing or damaged component with the spare.
- If a spare component is not available in inventory, or if the missing or damaged item is one of the radioactive standards or matrix drums, the PDP standards custodian, through or in conjunction with the assay coordinator, will immediately notify the NDA PDP coordinator. The SPT shall secure all materials and await further instructions.
- The PDP standards custodian shall insert each standard into the identified position of the matrix drum insert fixture as delineated on the sample configuration form. Source positioning shall be verified by the PDP standards configuration attestant as correct and documented.
- Once all standards have been verified as properly positioned within the insert fixture and placed in the correct matrix drum insert tube, the PDP standards configuration attestant shall thread the security lanyard through all the insert fixture TID rings and seal the PDP sample with the appropriate serialized TID.
- The PDP standards custodian shall seal the envelope containing a copy of the completed PDP sample configuration form (see Appendix D) with a tamper-indicating security seal and affix it to the PDP sample drum. A site-specific

sample information form may accompany this copy of the PDP sample configuration form if a specific form is required by the site staff responsible for SNM accountability. The sealed sample configuration form provides relevant PDP standard information, including standard activity and standard location within the matrix drum. The configuration form may be opened only during an emergency or at the time of sample disassembly. If the security seal for the PDP sample configuration form is broken before PDP sample disassembly at the conclusion of the cycle, the NDA PDP coordinator is to be notified and analysis data for that sample may be considered invalid.

5. The SPT shall return any unused PDP materials to storage and secure the PDP standards storage area with a TID. A site-specific security system may be used in place of the supplied TID as long as the prevention of unauthorized access to the unused standards or storage-related documentation can be ensured and documented.
6. The SPT shall prepare a PDP Sample Custody Form for Nondestructive Assay (see Appendix D) for transfer of the sample to the measurement facility. The PDP standards custodian shall transfer the PDP samples and custody forms to the assay coordinator by obtaining the assay coordinator's signature for each prepared PDP sample.

If multiple assay systems are to be qualified at one site or facility, it is the assay coordinator's responsibility to oversee and manage schedules and PDP sample transfers between the various assay systems at the site. If there is not enough time to make all measurements for the number of participating assay systems, the assay coordinator should request an extension pursuant to Section 5.2.

The SPT and/or assay coordinator shall maintain all records of PDP sample preparation in strict confidence until CBFO distributes a final report or as notified by the NDA PDP coordinator.

## **5.0 ANALYTICAL AND DATA REPORTING REQUIREMENTS**

This section describes required activities for NDA PDP sample acceptance, analysis, reporting, and return to the SPT when measurements are complete.

### **5.1 NDA PDP Sample Receipt and Custody**

The participating measurement facility assay coordinator is responsible for accepting PDP samples, ensuring adherence to chain-of-custody protocols, and confirming the accuracy of each sample custody form.

On initial receipt of a PDP sample, the assay coordinator shall inspect the condition of the sample seals by checking the TIDs and ensuring they are intact. If a problem exists with the integrity of a TID, the PDP assay coordinator shall not accept the sample and shall notify the SPT.

Provided that the TIDs are intact and the sample custody forms are properly completed, the assay coordinator shall review, sign, and date the sample custody form. This custodial signature means that the measurement facility accepts the PDP sample for analysis. The SPT makes and retains a copy of the custody form for each sample.

All subsequent transfers of the PDP sample drums within the measurement group and ultimate return to the SPT shall be documented on the PDP sample custody form accompanying the PDP sample. The final signature on the custody form is to be the SPT of the PDP sample at the conclusion of assay system measurements.

### **5.2 Analysis**

The measurement facility shall perform six analyses of the contents of each PDP sample using the assay procedures that are implemented and approved for use in the WIPP waste characterization program. These procedures must have been reviewed and approved following CBFO protocol. The PDP sample must be completely removed and replaced between each sequential measurement. The PDP sample shall be analyzed six times only. It is not permissible to select six measurements from a larger measurement set and submit them as the PDP cycle measurement replicate set. Analyses should be completed and reported as soon as possible, except in cases where one or more of the six replicates are judged by the NDA analyst to be defective due to instrument failure or operator error. In this case, the NDA PDP coordinator is to be notified. Based on circumstances, the NDA PDP coordinator may give permission to re-run the six-replicate set prior to submitting the measurement report sheets.

If a participant's analyses will not be reported by the due date, the CBFO and the NDA PDP coordinator must be notified in writing (e-mail is acceptable) as soon as possible, and the participant must request a time extension. Extensions must be requested before the due date, and will be granted or rejected in writing by the CBFO. If an extension has not been requested prior to the due date, the NDA PDP coordinator may make the actual identity and concentration of the content of the PDP samples at any time thereafter. Any participant that has not yet reported will be unable to submit the data for scoring in that PDP cycle, unless other arrangements have been made with the NDA PDP coordinator.

### **5.3 Reporting**

The participating measurement facility report shall contain those isotopes identified from the list in Section 1.4, for each replicate analysis. The activity of each isotope detected and quantified must be reported. If the test sample is below the minimum detectable concentration (MDC) or outside the

calibration range of the measurement system, measured values are not required in the report, only the reason for no value (i.e., <MDC).

A signed measurement report for each replicate analysis of each PDP sample shall be forwarded directly to the NDA PDP coordinator. All analytical reports must be received by the NDA PDP coordinator no later than 28 calendar days after receipt of instructions (except as noted in Section 5.2).

### 5.3.1 Report Contents

To submit PDP sample measurement results, an NDA PDP report form is required for each individual replicate analysis. The Performance Demonstration Program Report Form - Nondestructive Assay (see Appendix D) should be used to report the data to the NDA PDP coordinator. Continuation sheets may be used if measurement facility comments exceed the allocated space. Completed forms shall be signed by a measurement facility staff member responsible for transfer of the results to the NDA PDP coordinator. Reports should contain any other sample measurement information deemed relevant by the measurement facility. Corrections to data, or data inadvertently omitted from the report form will be accepted if the PDP coordinator is notified before the cycle scoring report is issued.

Each NDA PDP report form must contain, at a minimum, the following information for each determination:

- Identification of the reporting measurement facility, including system ID obtained from the NDA PDP registration process
- Identification of the PDP cycle for which the data are being reported
- Identity of the matrix drum by serial number from the PDP Sample Custody Form for Nondestructive Assay
- Identification of the measurement system and method (including software/hardware configuration version) used for each isotope (measurement systems using constant isotope ratios are to be indicated on the report form)
- Identification of the PDP sample measurement replicate number corresponding to the analytical data
- Identity and activity in curies for each isotope identified and quantified
- Total uncertainty for each identified isotope at one standard deviation
- Total <sup>239</sup>Pu fissile gram equivalent (g) and associated total uncertainty at one standard deviation
- Total TRU alpha activity and associated total uncertainty (curies) at one standard deviation
- Thermal power and associated uncertainty at one standard deviation (W)
- Elapsed counting time
- Date and time of sample analysis.

### 5.3.2 Analytical Records

Records generated by the NDA PDP Program and participating sites during the conduct of a PDP cycle are QA records. All NDA PDP cycle documentation must be maintained in a traceable and auditable

condition. Storage conditions and duration must meet the requirements of the QAPD and other implementing QA documents and procedures.

## **5.4 Completion and Disassembly**

After the replicate measurement sets are complete and the PDP samples are returned from the assay coordinator, the SPT is authorized to disassemble the PDP samples at the site's convenience. The PDP samples shall be disassembled in the following manner:

- The PDP standards custodian shall retrieve the appropriate PDP sample custody form for nondestructive assay and the PDP sample configuration form for each PDP sample to be disassembled (see Appendix D).
- The PDP standards custodian shall determine the condition of the TID that seals the copy of the PDP sample configuration form (see Appendix D) attached to the sample drum. If the seal is broken, the PDP standards custodian shall contact the assay coordinator, who will evaluate and notify the NDA PDP coordinator. If the seal is not already broken, the PDP standards custodian will break the security seal and remove the PDP sample configuration form from the PDP sample drum. If the security seal for the PDP sample configuration form is compromised before PDP sample disassembly, all analysis data for that sample may be considered invalid. In such an event, the PDP standards custodian shall notify the NDA PDP coordinator through or in conjunction with the facility assay coordinator for determination of measurement data validity.
- The PDP standards custodian shall determine the condition of the TID on the drum lid locking bolt that prevents tampering with the matrix drum source insert fixtures. The PDP standards custodian will break the TID and remove the security lanyard, allowing the insert fixtures containing the PDP standards to be removed (see Appendix D). If the TID is broken before PDP sample disassembly, the PDP standards custodian shall notify the NDA PDP coordinator through or in conjunction with the assay coordinator for determination of measurement data validity. If any TID, custody seal, matrix drum, or standard shows evidence of tampering, the PDP standards custodian shall ensure that such evidence is secured and that the condition is noted on the custody form.
- The PDP standards custodian shall remove each PDP standard from the source insert fixture. Each SPT member then independently verifies that the source positioning is correct by initialing the PDP sample configuration form. If there is a damaged, missing, or misplaced PDP sample component, this information must be reported to the assay coordinator, who will evaluate and notify the NDA PDP coordinator.

Once all PDP standards have been removed, the standards custodian will coordinate the return of the PDP matrix drums and the PDP sample standards to the designated, secured storage area using the facility's normal storage procedures. At completion of the disassembly operations, the PDP sample custody form shall be noted as "Disassembled." The original signed NDA PDP Sample Configuration Form and the NDA PDP Sample Custody Form are required QA records and must be returned to the NDA PDP coordinator.

## 6.0 EVALUATION OF PERFORMANCE DATA

In the NDA PDP measurement system performance is evaluated using the parameters precision and bias. Precision is defined as the percent relative standard deviation (%RSD), the standard deviation of the six sequential replicate measurements divided by the known value times 100. Bias is the systematic error component of the total measurement uncertainty determined as the ratio of the mean of the measurement replicate set to the known value times 100 or the percent recovery (%R).

Identified  $\alpha$ -activity ranges to which the NDA PDP quality assurance objectives (QAOs) apply for the PDP sample are listed in Table 2. See Appendix E for explanation of the statistical basis for the measured precision QAOs. The basis for the Table 2 bias acceptance bounds as determined from the six replicate measurements is also discussed in Appendix E.

The scoring system for the PDP is pass-fail. In order to pass a PDP test cycle, the measurement results must fall within the Table 2 test criteria. In order to pass the NDA PDP cycle, the measurement system must successfully comply with both the precision and bias criteria per Table 2. Both precision and bias are measured for all NDA PDP samples. Different criteria have been established for PDP drum matrices called noninterfering and interfering. Precision and bias for the noninterfering matrix are determined from measurements on the noninterfering (empty) matrix drum. Precision and bias determinations for all NDA PDP drums containing waste matrices are compared to the criteria for interfering matrices.

Table 2. PDP activity ranges and associated quality assurance objectives.

Activity range	Range of sample activity in $\alpha$ -curies <sup>a</sup>	QAO for precision <sup>b</sup> (%RSD)	Maximum Measured Precision <sup>c</sup>		Bias <sup>d</sup> (%R <sub>L</sub> and %R <sub>H</sub> )	
			Noninterfering matrix (%RSD)	Interfering matrix (%RSD)	Noninterfering matrix (%R)	Interfering matrix (%R)
Low	> 0 to 0.02	29.2%	14%	16%	Low: 70% High: 130%	Low: 40% High: 160%
Mid-Low	> 0.02 to 0.2	21.9%	10.5%	12%	Low: 70% High: 130%	Low: 40% High: 160%
Mid-High	> 0.2 to 2.0	14.6%	7%	12%	Low: 70% High: 130%	Low: 40% High: 160%
High	> 2.0	7.3%	3.5%	6%	Low: 70% High: 130%	Low: 40% High: 160%

%R = percent recovery

%RSD = percent relative standard deviation

a. Applicable range of TRU activity contained in a PDP sample; units are curies of alpha-emitting TRU isotopes with half-lives greater than 20 years.

b. Limits for one relative standard deviation,  $S/\bar{X}$ , expressed as a percent.

c. Measured precision that must be met to satisfy the precision criteria at the 95% upper confidence bound, based on six replicates. The values are one relative standard deviation referenced to the known value for the test.

d. %R<sub>L</sub> and %R<sub>H</sub> values used in Equation 3 to determine the 95% confidence bound for the ratio of the mean of the measured values to the known value, expressed as a percent.

The objective of the NDA PDP scoring system is to ensure that the QAOs for precision and bias are satisfied at the 95% confidence level for a reasonable number of replicate samples. A reasonable number of samples, as defined for the NDA PDP is six replicates. Setting the number of replicates at six is a compromise between minimizing facility operational impact through use of fewer samples and using a larger number, which increases the power of the test at the 95% confidence level. Therefore, six is the defined number of contiguous measurements to be performed and documented using the report forms. Six sequential replicates are all that are acceptable; multiple measurements from which six replicates are selected for reporting is not allowed.

## 6.1 Scoring System

Because NDA is inherently a probabilistic process, the specification of a scoring system to demonstrate compliance with the QAOs must be based on probabilistic confidence limits and bounds. The underlying distribution of any NDA measurement is assumed to be normal. However, the variance of this normal distribution, which is the true precision of the NDA instrument, is *a priori* unknown, and is one of the performance parameters addressed through the NDA PDP scoring system.

The measurement system precision is equal to the standard deviation of the underlying measurement distribution. The precision is estimated by making six replicate measurements on a single known PDP sample. The measured standard deviation is generally not identical to the underlying distribution standard deviation, but the two are related by the chi-square distribution. A chi-square distribution is assumed for the evaluation of precision. Similarly, the measured or the sampled mean will be related to the mean of the underlying distribution by the Student's *t*-distribution, because the underlying variance is not known.

To evaluate precision and bias, two parameters must be specified, the required confidence level, and the number of replicates. Because only six sample measurements are used in the NDA PDP, the numerical criteria for both the precision and the bias are adjusted to the 95% confidence level. Compliance with the NDA PDP QAOs requires demonstration at the 95% confidence level.

Precision is expressed as the measured %RSD for the PDP sample. Precision, as used in the NDA PDP, is calculated relative to the known total TRU  $\alpha$ -activity value of the PDP sample. This permits the test of precision to be equitable among the measurement systems and independent of the measurement bias. Referencing the standard deviation to the known value also preserves the assumption of the chi-square definition integral to the statistical basis as delineated in Appendix E. For the NDA PDP, the %RSD is determined using Equation 1.

$$\% \text{ RSD} = 100 \times \frac{\sqrt{\frac{\sum_{i=1}^n (x_i - \bar{x})^2}{n - 1}}}{\mu_0} \quad (1)$$

where:

- $x_i$  = replicate sample value
- $n$  = the number of measurements
- $\mu_0$  = actual known PDP sample value
- $\bar{x}$  = replicate set average, defined by



$$\bar{x} = \frac{\sum_{i=1}^n x_i}{n} . \quad (2)$$

The measurement precision QAO limit for the noninterfering matrix is calculated from the assumption that the PDP precision criteria represent the 95% upper confidence bound of a chi-squared distribution at five degrees of freedom. Column 4 of Table 2 shows the resulting measurement precision limits for the noninterfering matrix as a function of activity range. The interfering matrix precision limits in column 5 of Table 2 were modified from the noninterfering, based on the additional sources of uncertainty associated with the interfering-type matrix drums.

For evaluation of measurement system bias, the Student's  $t$ -distribution  $t_{0.975}$  percentile value is used in conjunction with the %RSD and the bounds in columns 6 and 7 of Table 2. Assuming six replicate samples and a 95% confidence level, this equation can be expressed as

$$(\%R_L + 1.049\%RSD) \leq \left( 100x \frac{\bar{x}}{\mu_o} \right) \leq (\%R_H - 1.049\%RSD) . \quad (3)$$

where:

- $\%R_L$  = low percent recovery limit specified in Table 2, column 6 or 7, as appropriate
- $\%R_H$  = high percent recovery limit specified in Table 2, column 6 or 7, as appropriate
- 1.049 = constant based on  $t_{1-\alpha/2}$  at  $\alpha = 0.05$  and the square root of the number of replicates.

Equation 3 requires that

$$1.049 \times \%RSD \leq | 100 - \%R_{L,H} | \quad (4)$$

Otherwise, the test will fail.

Appendix E provides a detailed discussion of the statistical bases for the precision and bias scoring criteria.

### 6.1.1 Interfering Matrix Bias

*Purpose:* NDA results for replicate analyses for PDP samples of known TRU alpha activity are used to determine the bias associated with measurement facility determinations of total TRU  $\alpha$ -activity. In this program, the bias is determined from the measurement results for PDP samples that include both variance and bias elements, including effects due to sample matrix configuration and nuclear material characteristics.

*Criteria:* The results reported for total TRU alpha activity shall not deviate from the reference value,  $\mu_o$  (known value), by more than the amount determined using Equation 3 in conjunction with the appropriate  $\%R_{L,H}$  values specified in column 7 of Table 2. The selection of the appropriate criteria specified in Table 2 is based on the known total TRU  $\alpha$ - activity range in which the prepared PDP sample falls.

*Method:* Measurement system bias shall be computed by measuring six sequential replicate samples and calculating the mean,  $\bar{x}$  (Equation 2) and the %RSD (Equation 1). The measurement will pass the

interfering bias QAO if Equation 3 is satisfied and will fail if Equation 3 is not satisfied. The values for %R in Equation 3 are the low and high values specified in the total bias column of Table 2 that correspond to the total TRU  $\alpha$ -activity range in which the prepared PDP sample falls.

*Actions:* For PDP samples for which the measured %R is outside the computed limits for the activity range tested, the measurement facility will be scored as failed for that specific activity range. The impact of exceeding an action level on overall measurement facility performance is given in Section 6.2. In accordance with Section 7, the site project manager is responsible for ensuring that appropriate corrective actions are taken.

### 6.1.2 Noninterfering Matrix Bias

*Purpose:* NDA results for replicate analyses for PDP samples of known TRU  $\alpha$ - activity in a noninterfering matrix drum are used to determine noninterfering measurement bias for total TRU  $\alpha$ -activity.

*Criteria:* The results reported for total TRU  $\alpha$ -activity %R shall not exceed the bounds as calculated from equation 3, using the values for %R<sub>L,H</sub> specified in column 6 of Table 2 (bias for noninterfering matrix). The selection of the appropriate criteria specified in Table 2 is based on the known TRU total  $\alpha$ -activity range in which the prepared PDP sample falls.

*Method:* The method for determining the measurement system bias shall be identical to the method for the bias determination of PDP samples (Section 6.1.1), except that the %R<sub>L,H</sub> values used in Equation 3 will be from the Table 2 noninterfering bias column. Also, the noninterfering bias evaluation applies only to PDP samples assembled using the noninterfering matrix drum or other drum approved by the CBFO as noninterfering.

*Actions:* The actions for the noninterfering bias determination are identical to the actions for the determination of the bias determination on interfering matrix drums (Section 6.1.1).

### 6.1.3 Precision of NDA PDP Test Samples

*Purpose:* To demonstrate compliance with the NDA PDP QAOs for precision, NDA measurement results from replicate analyses of a PDP sample of known total TRU  $\alpha$ -activity are used to determine measurement system precision for total TRU  $\alpha$ -activity.

*Criteria:* The results reported for total TRU  $\alpha$ -activity from replicate measurements of a PDP sample shall not exceed the allowable %RSD per Table 2, column 4 for the noninterfering matrix drums, and column 5 for the interfering-type PDP matrix drums.

*Method:* The analytical results from the six replicate measurements of a PDP sample are used to calculate the %RSD using Equation 1. The measured %RSD is compared with the applicable limits listed in Table 2. For the noninterfering matrix drum, if the %RSD value determined from the measurement data is less than the range-specific value in Table 2, column 4, the measurement system passes the test. For the interfering PDP matrix drum, if the %RSD value determined from the measurement data is less than the range-specific value in Table 2, column 5, the test is passed.

*Actions:* For any sample for which results exceed the appropriate QAO for precision in any sample  $\alpha$ -activity range, the measurement facility will be scored as failed for that specific total TRU  $\alpha$ -activity range. The impact of exceeding an action level on overall measurement facility performance is given in

Section 6.2. In accordance with Section 7, the site project manager is responsible for ensuring that appropriate corrective actions are taken.

## **6.2 Use of NDA PDP Test Performance to Support WIPP Qualification**

The primary purpose of the NDA PDP is to independently acquire data on measurement system capability, as well as to uncover technical or quality assurance-related deficiencies that may impact the characterization of WIPP wastes. Through implementation of the NDA PDP, information is gathered on system performance, NDA facility management, NDA procedural strategies, corrective action effectiveness, and data quality assurance. Although the NDA PDP scoring criteria and associated QAO limits and bounds are precisely defined relative to test “pass” or “fail,” the CBFO may make use of other information derived from the NDA PDP program and/or other sources in their final assessment of measurement facility capability, performance, and approval status.

The NDA PDP plan requires a measurement facility to have in place approved NDA system procedures for the analysis of WIPP wastes. The procedures and measurement system parameters used to analyze WIPP wastes are to be the same as those used in the analysis of NDA PDP samples. Successful completion of an NDA PDP cycle for a given measurement system requires that data reported by the facility and evaluated per the NDA PDP bias and precision scoring routines be within applicable limits and bounds for each PDP sample.

Based on strict interpretation of the scoring criteria and Table 2 limits, to pass a NDA PDP test cycle, measurement system report data must yield both bias and precision values that are in the acceptance regions for each PDP sample. Implied in this interpretation is that a measurement system with a pass status based on a PDP sample consisting of a combustibles matrix and a total TRU  $\alpha$ -activity in the mid-low activity range ( $>0.02$ – $0.2$  Ci) is qualified to measure actual WIPP combustible-type wastes with a total TRU  $\alpha$ -activity in the mid-low range. In reality, a pass result on a this combustibles PDP sample allows the measurement facility to analyze WIPP wastes that are similar to combustibles in terms of matrix composition and configuration through a total TRU  $\alpha$ -activity range for which the measurement system has been approved through other methods such as independent technical reviews.

The CBFO has considerable latitude in overall measurement system approval, disapproval, or application restrictions based on NDA PDP results. For example, if information exists from sources other than the NDA PDP, indicating that the MDC for a given measurement system is higher than a certain value, it may limit the analyses of WIPP waste, to activities that exceed a certain value regardless of NDA PDP results. In a like manner, the CBFO may allow a given system to process WIPP wastes even though it failed one or more of the NDA PDP criteria on one or more PDP samples. An example of such an allowance would be that the PDP test sample was not representative of the measurement facility’s waste inventory. Another example may be that a particular NDA system would have an unusually high bias for a test sample configured in a particular manner. Although other NDA system types may process such a sample with a pass status, there may be some systems with inherent response characteristics that have a lesser probability of passing. Provided a reasonable technical basis can be established for an NDA PDP failure, the CBFO has the latitude to waive such per the circumstance. This is consistent with a previously noted purpose of the NDA PDP. It is a part of an overall assessment and approval process for characterization of WIPP wastes and not the definitive qualifier.

Due to the limited number of the tests within a PDP cycle, a failure must be assessed thoroughly to determine the extent of impact on a site’s TRU waste characterization program. In many cases, a failure involves the ability of an assay system to measure one or a limited number of similar waste streams. Alternatively, depending on the specific nature of the test and/or failure that occurred, the failure could indicate a broader systemic problem affecting all measurements made by the system. Such issues must be

considered by the NDA PDP coordinator and the CBFO when identifying any “potentially affected assay data” following a PDP failure.

The CBFO may elect to grant or deny approval regardless of NDA PDP results, provided an adequate technical basis exists. The CBFO may also grant approval based on certain conditions due to the results of a given NDA PDP cycle and other circumstances. For those cases where approval, conditional or otherwise, is **not** given by the CBFO based on the results of the NDA PDP and/or other information, the measurement facility must:

1. Based on review comments provided by the CBFO, identify the basis of the failure and develop a corrective action plan (CAP) that addresses all contributing components, technical and/or administrative.
2. Submit the CAP to the CBFO and resolve all issues resulting from the review. The CBFO must approve the CAP prior to implementation by the measurement facility.
3. Implement the CBFO-approved CAP and provide evidence that the plan effectively addresses the deficiency.
4. Obtain final CBFO approval, conditional or otherwise, to process WIPP wastes with the NDA characterization modifications.
5. Identify all radioassay data potentially affected by the deficiency. Evaluate and develop a plan for disposition of radioassay data potentially compromised by the deficiency. Such data shall be treated as potentially nonconforming under the facility’s QA program.
6. Submit a nonconforming radioassay data disposition plan to CBFO for review and approval. Implement the plan as approved by the CBFO.

## **7.0 REPORTING PERFORMANCE DATA**

### **7.1 Summary of Data**

The NDA PDP coordinator shall review, evaluate, and score the reported data results, compile them into a master summary, and deliver this summary to the CBFO within four weeks after final site data are received. The summary shall include the values reported by the measurement facilities, the reference activity values, the acceptance ranges, and the pass-fail status of each individual measurement system, in addition to CAP status, as applicable.

The CBFO, with the assistance of the NDA PDP coordinator, will evaluate individual measurement facility performance and approve individual measurement systems for participation in the WIPP waste characterization program. At any point in the evaluation process, the CBFO may request additional information from, or hold discussions with, participants regarding systems and procedures used to perform calibrations, performance check samples, PDP samples, and actual wastes. As a result of the PDP performance and related evaluations, the CBFO may issue unqualified approvals, may disapprove a method, may issue approvals limited to specific activity, waste weight ranges or waste types, or may issue conditional approvals on other required actions. The generator site project managers shall be responsible for ensuring that appropriate corrective actions are taken and that all conditions and limits on method approvals are met.

### **7.2 Distribution of Reports**

Copies of the summary report are distributed to each of the DOE Operations Offices involved, each of the participating measurement facilities, and other individuals and organizations deemed appropriate by the CBFO. The CBFO shall also provide written notification to the DOE Operations Offices regarding the adequacy and approval status of their participating measurement facilities. Distribution will be in an electronic format unless the recipient requests a hardcopy report.

### **7.3 Supplemental NDA PDP Cycles**

A supplemental NDA PDP cycle can be requested by measurement facilities that have failed one or more NDA PDP samples, for a newly implemented system, or for a system that has been inactive or has undergone changes which could impact the NDA PDP approval status. Requests must be submitted in writing to the CBFO, accompanied by the information required in Section 6.2, as applicable. If CBFO authorizes a supplemental cycle, the schedule of cycle initiation, analysis, scoring, and approval/disapproval actions by CBFO will be specified for each supplemental cycle. The schedule should consider a review of impacts on the overall WIPP schedule and program costs, and may include discussions with the potential participants. Timing and selection of measurement facilities for participation in supplemental cycles will be at the discretion of the CBFO. Costs of supplemental cycles requested by the measurement facility are typically borne by the requesting facility.

### **7.4 Measurement Facility Status**

Once the CBFO has determined measurement facility status as “approved” with respect to analyses that are required to demonstrate compliance with regulatory requirements, such status shall remain in effect for a maximum of 13 months (12 months plus a one-month “grace” period). All measurement facilities must participate in the annual primary cycle in order to remain qualified to perform WIPP analyses. Measurement facilities obtaining approved status through a supplemental cycle must participate in the next regular primary cycle to maintain their approved status. Treatment of radioassay data by facilities undergoing a change in status is discussed in Section 6.2.

The qualification period for a measurement facility begins with the date that analyses in a PDP cycle are completed. At the end of the 13th month, a facility that has not yet successfully completed their analyses of PDP samples to re-qualify their method may choose to proceed at risk with WIPP analyses, or to cease operations. The facility should recognize that data obtained at risk may be found to be unacceptable to WIPP. Data generated at risk cannot be used for characterizing waste for shipment to WIPP until:

- The system used to collect the data passes the NDA PDP, and
- The data have been reconciled through the disposition of a nonconformance report.

## **7.5 Quality Assurance Records**

The minimum QA records for the NDA PDP are identified and listed below in accordance with the QAPD requirements. In addition, the NDA PDP coordinator may determine that records of other program activities are QA records and enter them into the QA records system with the same level of control and maintenance.

These QA records may be organized by NDA PDP Plan revision, by PDP cycle, or other principle, as applicable. These records are nonpermanent records and shall be maintained in accordance with the QAPD requirements. Records disposition, when applicable, will be in accordance with CBFO/OCT requirements, approved procedures, and work plans.

All QA records identified in this plan shall be stored in accordance with record storage requirements in the QAPD. Access to QA records will be limited to personnel involved in the program or having related QA or records custodial responsibilities.

## 8.0 QA RECORDS FOR THE NDA PDP

The following documents will be maintained as QA records for the NDA PDP:

- Work plans (all revisions)
- PDP plans (all revisions)
- Procurement records
- Radioactive standard and matrix drum design and production records (each drum and PDP standard production phase)
- SPT training (training materials and attendance records)
- Assay system registration forms
- Records of cycle set-up (notification letters, shipping records, and other correspondence)
- Participant assay reports and supporting forms (assay data report forms, chain-of-custody records, and configuration forms)
- Scoring reports
- Reviews of corrective actions and supporting data and recommendations made to CBFO.

The following matrix of QA records provides a more detailed listing of records and designates responsibility for maintenance.

### NDA PDP Documentation/Records List

Document(s)	QA Record	Storage Location	Comments
1. PDP Plan revisions	Yes	CBFO	For each revision.
2. PDP Plan revisions — comments and resolutions	Yes	CBFO	For each revision.
3. SPT training packages	Yes	CTAC	For each revision.
4. Approval from CBFO for schedule and participants	Yes	CBFO	Letter or hard copy of e-mail(s)
5. Participant NDA PDP program documentation <ul style="list-style-type: none"> <li>- Procedures</li> <li>- Raw data</li> <li>- Calibration records</li> <li>- Training records</li> </ul>	Yes	Participant	These should be exactly the same as are maintained/generated in waste characterization activities; required by WAC and PDP Plans.
6. SPT training records <ul style="list-style-type: none"> <li>- Completion forms</li> </ul>	Yes	Participant	Copies to CBFO and CTAC

Document(s)	QA Record	Storage Location	Comments
7. Correspondence to participants providing instructions for execution of an NDA PDP cycle	Yes	CBFO	Copies to CBFO and CTAC.
8. Sample configuration instructions accompanying notification	Yes	CBFO	Developed by the NDA PDP coordinator. Confidential until completion of cycle.
9. Participant correspondence requesting time extension for submitting results	Yes	CBFO	As required by PDP Plans, e-mails to the NDA PDP coordinator.
10. Responses to extension requests	Yes	CBFO	Responses issued by CBFO.
11. Participant data packages with cover letters (including any revisions and corrections to data) -Assay data report forms -Chain-of-custody records -Configuration forms -Disassembly forms	Yes	CBFO	Formally transmitted to CBFO on completion of all cycle-specific activities.
12. Scoring report	Yes	CBFO	Formally transmitted to CBFO/CTAC file copy. CBFO approves distribution by the NDA PDP Coordinator.
13. Records of QA review of scoring report	Yes	CBFO	Formally transmitted to CBFO/CTAC file copy.
14. Letters issued by CBFO to provide status notification to participants	Yes	CBFO	CTAC maintains a file copy.
15. Transmittal letter with cycle specific records	Yes	CBFO	Transmitted upon completion and receipt of all cycle records. .
16. Current document, software revision list, and software verification and validation documentation	Yes	CBFO	Required by the CBFO QAPD.
17. NDA PDP MP	Yes	CTAC	CBFO reviews to ensure MP meets CBFO requirements.
18. Participants' reports of corrective actions	Yes	CBFO	Submitted to CBFO.



## 9.0 GLOSSARY

**ACTIVE SYSTEM** – An operational NDA system currently located at a participant site and qualified to perform waste characterization activities at that site. This includes systems that are not actively assaying waste because of scheduling issues or other site issues not connected to system capability or readiness.

**ASSAY COORDINATOR** – Facility contact responsible for accepting PDP samples and ensuring that chain-of-custody protocols are followed.

**BIAS** – The systematic error component of the total uncertainty; that is, a constant positive or negative deviation of the method average from the correct value or an accepted reference value under specific measurement conditions. Bias is computed as %R.

**CORRECTIVE ACTION** – Measures taken to rectify conditions adverse to quality and where necessary, to preclude their recurrence.

**INACTIVE SYSTEM** – A CBFO-approved and operational NDA system not being used to characterize WIPP wastes for the OCT at the time a primary PDP cycle is conducted.

**MATRIX DRUM** – 208-liter (55-gallon) steel drum designed, fabricated and serial-numbered for the PDP, containing a surrogate waste matrix configuration representative of a specified population of the actual DOE waste inventory. The interfering or noninterfering matrix drum contains an internal structure for insertion and positioning of PDP standards and a 90 mil polyethylene liner.

**NDA PDP COORDINATOR** – An individual responsible for coordinating the technical operations of the NDA PDP, including PDP sample component preparation, SPT oversight, scheduling, scoring, and report summary generation.

**NONDESTRUCTIVE ASSAY** – Assay methods for waste items that do not affect the physical or chemical form of the material.

**NONINTERFERING MATRIX** – A matrix drum that contains only the supports for insertable standards and 90 mil polyethylene liner that serves as the noninterfering matrix test sample.

**PDP MANAGER** – An individual responsible for overall performance of the NDA PDP.

**PDP SAMPLE** – A blind test sample prepared and sealed by the SPT for subsequent analysis by a measurement facility for qualification under the PDP. A PDP sample for the NDA PDP is comprised of a 55-gallon matrix drum and insertable PDP standards. Sample matrix and source characteristics will representatively span nominal waste characteristics including, but not limited to, isotopics, plutonium concentration, ( $\alpha$ ,n) reactions, interfering matrices, and source distribution.

**PDP STANDARD** – A radioactive source specifically prepared or acquired and certified for the NDA PDP.

**PDP STANDARDS CONFIGURATION ATTESTANT** – A member of the two-person SPT responsible for verifying the proper emplacement of PDP sample standards and performing sample security-related procedures.

**PDP STANDARDS CUSTODIAN** – The lead member of the SPT responsible for coordination of on-site PDP sample preparation activities.

**PRECISION** – A measure of the variance among individual measurements of the same property made under prescribed conditions; expressed as a percent relative standard deviation (%RSD).

**PRIMARY CYCLE** – The annual NDA PDP cycle.

**PROGRAM COORDINATOR** – A CBFO-designated organization that administers and coordinates PDP functions. The program coordinator will designate the PDP manager.

**SAMPLE PREPARATION PROCEDURE** – A procedure generated by the NDA PDP coordinator for each measurement facility for each PDP cycle. This procedure provides instructions to the SPT on PDP standard placement within a specified PDP matrix drum.

**SAMPLE PREPARATION TEAM** – A two-person team, consisting of a PDP standards custodian and PDP standards configuration attestant that prepare and certify measurement facility PDP samples. The SPT is responsible for ensuring that each PDP sample is prepared according to the PDP procedures. In addition, the SPT ensures proper disassembly and return to storage of all PDP components after analysis by the measurement facility.

**SUPPLEMENTAL CYCLE** – An NDA PDP cycle in addition to the annual primary PDP cycle. Reasons that a supplemental cycle may be necessary include accommodation of measurement facilities unable to participate in the primary cycle, to support implementation of new or modified systems, or to perform specific testing as directed by the CBFO. Additional supplemental cycles may be conducted on an as-needed basis at CBFO direction.

**TOTAL UNCERTAINTY** – The propagated measurement error from all bias and precision sources including interference effects such as variable matrices, isotopic compositions, spatial distributions, contaminating radionuclides, and others.

## 10.0 REFERENCES

*Transuranic Waste Baseline Inventory Report*. DOE/CAO-95-1121, current revision, U.S. Department of Energy Carlsbad Field Office, Carlsbad, New Mexico.

*Waste Acceptance Criteria for the Waste Isolation Pilot Plant*, DOE/WIPP-02-3122, current revision, Waste Isolation Pilot Plant, U.S. Department of Energy, Carlsbad, New Mexico.

*Quality Assurance Program Document*. CBFO-94-1012, current revision, U.S. Department of Energy Carlsbad Field Office, Carlsbad, New Mexico.

*Performance Demonstration Program Plan for Nondestructive Assay of Boxed Wastes for the TRU Waste Characterization Program*, DOE/CBFO-01-1006, current revision, U.S. Department of Energy Carlsbad Field Office, Carlsbad, New Mexico.

*Performance Demonstration Program Management Plan*, DOE/CBFO-01-3107, current revision, U.S. Department of Energy Carlsbad Field Office, Carlsbad, New Mexico.

NDA PDP Working Reference Material Productions Plan – Phase I, LAUR-96-2277, current revision, Los Alamos National Laboratory, Los Alamos, New Mexico.

NDA PDP High Mass Working Reference Material Production Plan – Phase II.A, CST-8-PLA-STD-106, LAUR-98-213, current revision, Los Alamos National Laboratory, Los Alamos, New Mexico.

*National TRU Waste Program Non-Destructive Assay Performance Demonstration Program Large Particle Working Reference Material Production Plan – Phase II.B*, LA-CP-01-208, current revision, Los Alamos National Laboratory, Los Alamos, New Mexico.

*National Transuranic Waste Program Nondestructive Assay Performance Demonstration Program Enriched Uranium Production Plan – Phase II.C*, LA-CP-00-54, current revision, Los Alamos National Laboratory, Los Alamos, New Mexico.

*National TRU Waste Program NDA PDP Depleted Uranium Production Plan – Phase II.D*, current revision, LA-CP-00-110, Los Alamos National Laboratory, Los Alamos, New Mexico.

*National TRU Waste Program Non-Destructive Assay Performance Demonstration Program Increased Americium: Plutonium Working Reference Material Production Plan – Phase III.A*, LA-CP-03-0072, current revision, Los Alamos National Laboratory, Los Alamos, New Mexico.

*National TRU Waste Program Non-Destructive Assay Performance Demonstration Program Heat Source Plutonium Working Reference Material Production Plan – Phase III.B*, LA-CP-03-0763, current revision, Los Alamos National Laboratory, Los Alamos, New Mexico.

*Design of Interfering Matrix Drums for the Non-Destructive Waste Assay Performance Demonstration Program for the National TRU Program*, INEEL/EXT-02-00320, February 2002, Idaho National Engineering and Environmental Laboratory, Bechtel BWXT Idaho, LLC, Idaho Falls, Idaho.

## **Appendix A**

### **Performance Demonstration Program for the Drum NDA PDP System Registration Form**

**Appendix A**  
**Performance Demonstration Program for**  
**Drum NDA PDP**  
**System Registration Form**

**General Instructions**

1. Registration forms are to be completed and returned to the NDA PDP coordinator at least 3 weeks prior to initial participation in the PDP.
2. Separate registration is required for each NDA measurement system.
3. After initial submission, the forms need be resubmitted only when there is a change in the registration information.
4. The NDA PDP coordinator will acknowledge all registration requests and assign a tracking identifier to each registered system.
5. The NDA PDP coordinator will maintain a current list of all registered systems.

**Instructions for Specific Questions**

Section A

1. Enter the full formal name of the measurement system.
2. Enter the acronym by which the system should be referenced.
3. Check the appropriate descriptor. "Fixed, Permanent" indicates that the system was intended to be installed permanently at the current location. "Transportable, Nonpermanent" indicates a long-term installation that can be relocated. "Mobile, Trailer" indicates systems intended for routine movement between sites for short-term contracts.
4. Enter the DOE site at which the system will be installed for the PDP cycle for which registration is being requested.
5. Enter the on-site location designator for the system.
6. Enter the name of the institution/facility/company that owns the system.
7. Enter the name of the institution/facility/company that operates the system.
8. Enter the name of the person who should be contacted for information on the system.
9. Enter the affiliation of the person who should be contacted for information on the system.
10. Enter the mail address for the person who should be contacted for information on the system.
11. Enter the express package delivery address for the person who should be contacted for information on the system.
12. Enter the e-mail address for the person who should be contacted for information on the system.

13. Enter the phone number for the person who should be contacted for information on the system.
14. Enter the fax number for the person who should be contacted for information on the system.

### Section B

1. Enter a description of the system, its principles of operation, and optional modes for assay.
2. Enter the identifier(s) for the written standard operating procedures (SOPs) that are used to operate the system for waste assay.
3. For each measurement mode of the system that may be used, enter the measurement principle (gamma, neutron), mode identification (active neutron, etc.), the procedure documenting selection criteria for selecting the subject modes, and the source of isotopic data used for that mode. Sources of isotopic data may be coded:  

P	=	isotopic data are measured as an integral part of the primary quantitative assay (e.g., gamma spectrometric methods used for both the quantitative and isotopic data).
S	=	isotopic data are derived from a secondary method (e.g., the primary quantitation is by passive neutron assay but isotopic ratios are obtained from an independent gamma spectrometric method).
AK	=	isotopic data are obtained from acceptable knowledge of the waste stream or container.

### Section C

- 1 through 5. Check off Yes or No to each question. For each "Yes," indicate the applicable mode(s) from section B.3.
6. Enter the possible mode(s) from section B.3 for each combination of activity range and waste type. Enter "NA" for combinations for which the system will not be used. If explanatory information is required, enter a number in the comments column and add the number and explanation to section D. For example, a system may have a calibration cut-off that falls at the midpoint of a test range. This may be indicated by accepting the range, but indicating a numerical limit in the comment.
- 7 through 9. Enter the values and units for any limits on PDP tests that, if exceeded, would prevent the system from assaying a PDP sample.

### Section D

Add any comments necessary to explain answers in any prior sections or supplemental information useful to the NDA PDP coordinator in planning effective PDP tests for the system. Attach continuation sheets as needed.

### Section E

Enter the requested information for the person submitting the registration form.  
Sign and forward the original of the form to the NDA PDP coordinator.

## Performance Demonstration Program for the Drum NDA PDP System Registration Form

<b>A. SYSTEM IDENTIFICATION</b>				
1. Official System Name:				
2. Acronym		Coordinator Use Only		
		System ID:		
3. Unit No. (this system):		Group No.:		
4. Mobility Type:	Fixed, Permanent <input type="checkbox"/> Transportable, Nonpermanent <input type="checkbox"/> Mobile, Trailer <input type="checkbox"/>			
5. Current Facility Location:		6. On-Site Reference		
7. System Owner:				
8. System Operator:				
9. Primary Contact Name:		14. E-Mail:		
10. Title:		15. Phone:		
11. Affiliation:		16. Fax:		
12. Postal Address:				
13. Express Package Address:				
<b>B. METHOD SUMMARY</b>				
1. Brief Description of Method:				
2. Associated SOP Identification(s):				
3. For each quantitative mode in which the system is used, complete the following:				
	3a. Assay Measurement Basis	3b. Mode	3c. Criteria for Selection	3d. Source of Isotopic Data
Quantitative Mode 1				
Quantitative Mode 2				
Quantitative Mode 2				
Quantitative Mode 3				

<b>C. SCOPE AND LIMITATIONS ON SYSTEM USE</b>						
			YES	NO	If YES, Mode No(s):	
1. Will the system be used to certify waste as TRU, for drums containing less than 0.01 curies of TRU isotopes?						
2. Will the system be used for wastes containing weapons grade plutonium?						
3. Will the system be used for wastes containing heat source plutonium?						
4. Will the system be used for wastes containing unknown isotopic ratios?						
5. Will the system be used for wastes containing uranium isotopes?						
6. Indicate the ranges and waste types for which the system intends participation in the PDP. For systems with multiple quantitative modes, be sure to indicate the applicable mode(s) for each case.						
Activity range	Range in alpha-curies <sup>a</sup>	Combustibles	Glass	Metals	Sludge	Comment No.
Low	> 0 to 0.02					
Mid-Low	> 0.02 to 0.2					
Mid-High	> 0.2 to 2.0					
High	> 2.0					
a. Range of TRU activity in a 55-gallon drum; units are curies of alpha-emitting TRU isotopes with half-lives greater than 20 years.						
7. Indicate the maximum activity permissible (including units) for testing the system due to calibration, radiological safety, or administrative limits:				Value:		Units:
8. Indicate the maximum weight permissible (including units) for testing the system due to calibration, safety, or administrative limits:				Value:		Units:
9. Indicate any other limitations on system tests: (Explanation in Comment No. _____.)				Value:		Units:
10. Did this system participate in any of the NDA PDP cycles 1 through 6C? If yes, which cycles and how was it identified?						
<b>D. COMMENTS</b> (Please add any information relative to participation of this system in the NDA Drum PDP)						
<b>E. Please register the system described in this application for participation in future cycles of the NDA Drum PDP. It is understood that system tracking, test design, approval conditions, participation requirements, and audit follow-up may be based on the information supplied in this application.</b>						
Typed Name:			Signature:			Date:
Phone Number:			E-Mail:			
<b>F. DISPOSITION</b> (coordinator use only)						



## **Appendix B**

### **NDA PDP Standard Encapsulation Design**

## Appendix B

### NDA PDP Standard Encapsulation Design

This appendix provides information on the NDA PDP standard encapsulation design used to contain the radioactive material/substrate mixture that constitutes a standard. Seven separate NDA PDP standard production phases were conducted at the Los Alamos National Laboratory. Each production phase was designed to yield a set of standards with specific attributes reflecting the DOE radioactive material inventory. All PDP standards use a dual cylinder encapsulation design and have identical internal and external dimensions. The encapsulation cylinders are fabricated from seamless stainless steel tubing for all production phases except Phase III.A, which is fabricated from seamless zirconium tubing.

The PDP standard encapsulation is comprised of an inner and outer cylinder. The bottom end cap of each cylinder is laser-welded in place. The inner cylinder is then filled with the appropriate mixture of radioactive material and substrate. Once the mixture has been packed to a height specified in the specification document, a graphite-felt frit is installed, which presses the nuclear material/substrate assembly in place when the top cap is inserted and attached. The graphite frit also ensures that the radioactive material/substrate configuration does not change over time. The inner cylinder top cap is then welded in place using the tungsten inert gas (TIG) method. After the top cap is welded in place, the inner cylinder is inserted into the outer cylinder and the top outer cylinder cap is TIG-welded in place. An elevation view of both the inner and outer encapsulation cylinders is shown in Figure B-1. The final assembled NDA PDP standard encapsulation unit is shown in Figure B-2. Prepared assemblies of the stainless steel and zirconium dual encapsulation, complete with the substrate matrix (no radioactive material) and helium fill, have been tested in accordance with ANSI/HPS N43.6-1997 and comply with ANSI Classification 97C43323.

The dimensional and material properties of the PDP standard were derived as a function of NDA PDP objectives, nondestructive waste assay system response characteristics, and practicalities of fabrication. A complete PDP standard specification with supporting analyses is provided in the Lockheed Martin Idaho Technologies Company document, *Performance Demonstration Program for Nondestructive Assay for the TRU Waste Characterization Program, Initial Cycle Source Design* (INEL-94/0104).

The as-specified PDP standard configuration complies with the following general requirements:

1. PDP standards must be physically stable and invariant with time in a defined geometry.
2. The PDP standard configuration must facilitate convenient loading of the standards into the PDP matrix drum.
3. The PDP standard dimensions must allow for the production of multiple-source spatial geometries within the PDP matrix drum.
4. The PDP standard encapsulation integrity must comply with all applicable standards and be acceptable for transportation to and storage at participating sites.
5. The PDP standard design must accommodate available fabrication technologies at a reasonable cost.

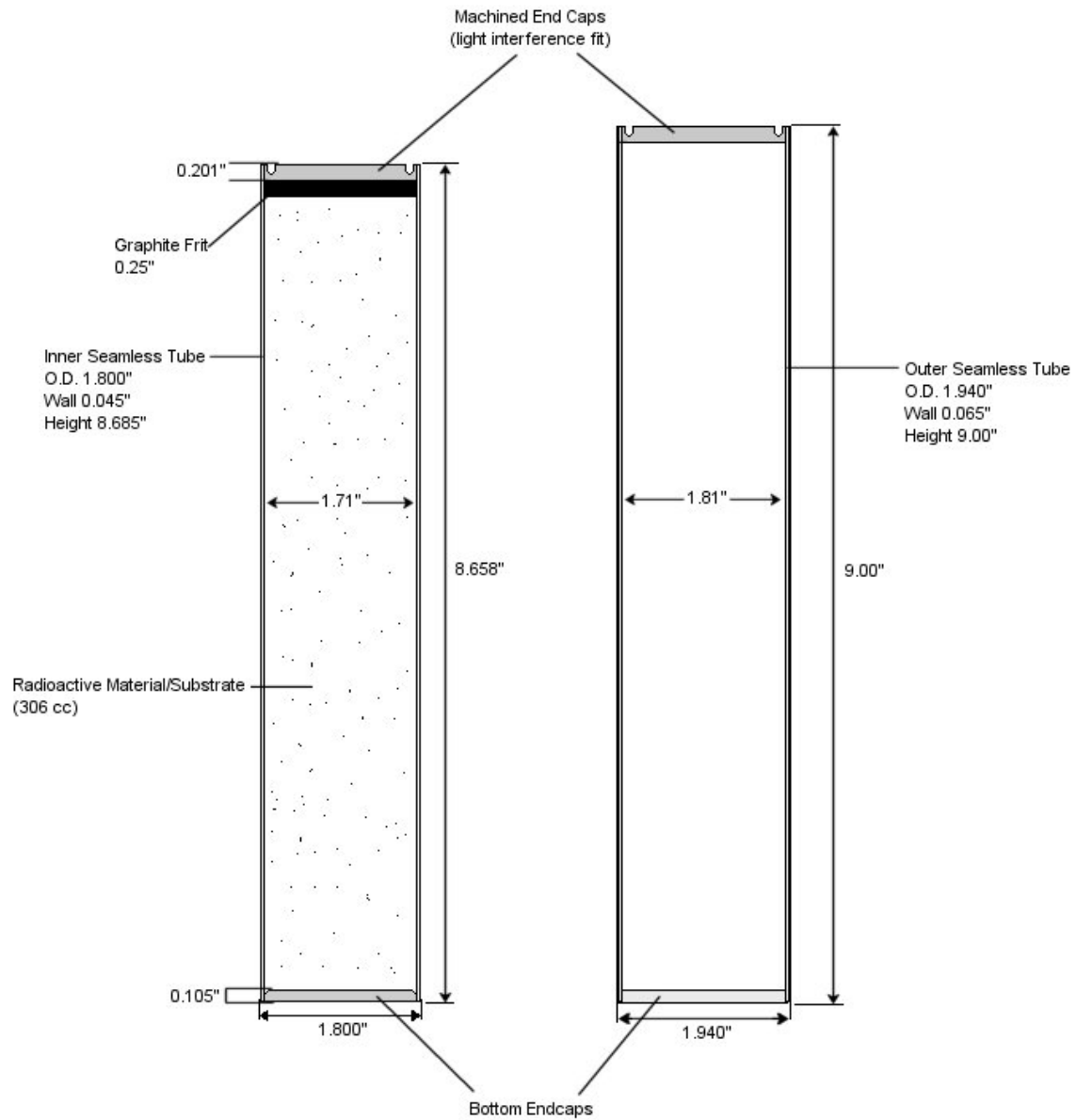


Figure B-1. PDP standard inner and outer cylinders, elevation view.

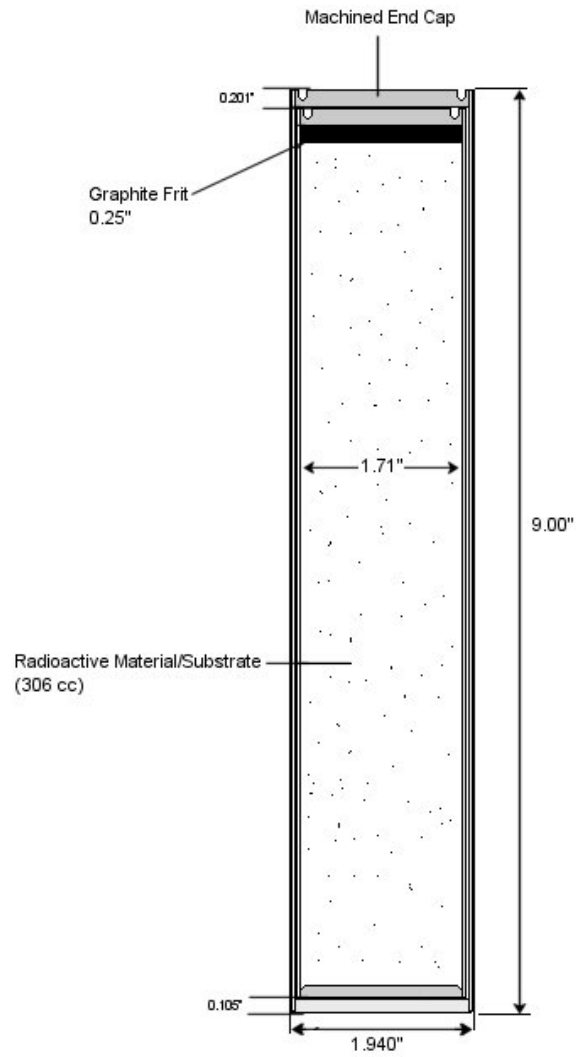


Figure B-2. PDP standard encapsulation assembly, elevation view.

## **Appendix C**

### **NDA PDP Matrix Drum Specifications and Descriptions**

## Appendix C

### NDA PDP Matrix Drum Specifications and Descriptions

This appendix provides an overview of the Nondestructive Assay (NDA) Performance Demonstration Program (PDP) matrix drum set, general specifications, design, and physical configurations. Additional technical detail on the design and fabrication of the NDA PDP matrix drum set is provided in the document *Design of Interfering Matrix Drums for the Non-Destructive Waste Assay Performance Demonstration Program for the National TRU Program*, INEEL/EXT-02-00320, February 2002.

Use of the waste matrix surrogate drums in combination with the NDA PDP standards provides the CBFO with information useful in assessing the ability of DOE facilities to meet DQOs for NDA of wastes intended for disposal at the WIPP. The CBFO uses data generated in the NDA PDP as part of the assessment and approval process for measurement facilities supplying services for the characterization of WIPP TRU waste.

The waste matrix types used in the fabrication of the NDA PDP matrix drum set are based on the major DOE inventory matrix types. The Baseline Inventory Report was used to identify the basic waste categories. These categories were subsequently collapsed based on the matrix attributes and characteristics to five waste types relative to NDA measurement technique and capability. The five waste types represented in the NDA PDP matrix drum set are: (1) noninterfering matrix (empty), (2) combustibles matrix, (3) inorganic sludge matrix, (4) metals matrix, and (5) glass matrix. Illustrations of the as-built construction of the five NDA PDP matrix drums are provided in Figures C-1 through C-5.

To position NDA PDP standard(s) within the matrix drum, insert fixtures are provided. The insert fixtures are fabricated from aluminum tubing as shown in Figure C-6. The NDA PDP standard is located at a given height within the fixture through the use of spring-loaded locating pins that snap into opposing holes in the insert fixture wall. When installed, the locating pin bisects the inner diameter of the insert fixture tube and provides a rest for the PDP standard. After the desired NDA PDP standard positions are achieved within the insert fixture, it is positioned into the matrix drum through a selected insert tube, also made of aluminum. In this manner, NDA PDP samples are configured as test samples for use in an NDA PDP cycle. The prepared NDA PDP sample must be secured with a TID to ensure the integrity of the test process and resulting data. Figure C-7 shows an NDA PDP sample with TID installed securing the insert fixtures within the matrix drum insert tubes.

General NDA PDP surrogate matrix drum specifications require the replication of the nominal waste matrix population parameters of interest, ensuring a stable non-variable matrix configuration and providing a convenient means to introduce and precisely locate PDP standards in the drum. Additionally, the NDA PDP matrix drum must conform to a set of requirements in order to support the needs of the NDA PDP Program. General specifications for an NDA PDP-type matrix drum are:

1. The design must provide a convenient means to externally introduce and locate PDP radioactive material standards (Appendix B) into the interior of a sealed 55-gallon PDP matrix drum. The design of this structure, called the internal support structure, must allow for positioning one or more PDP standards at internal drum radii and vertical heights sufficient to produce radioactivity distributions useful in performance assessments and instrument response studies. The insertion scheme and mechanism must allow for precise and reproducible positioning of standards.
2. The internal support structure used for PDP standard insertion and positioning shall also serve as a fixture allowing installed matrices to be fastened with coordinates oriented to a known reference point.

3. The internal support structure design and materials must inherently present minimal interference to the various waste NDA techniques.
4. The surrogate matrix drum design must support the assessment of NDA system function and performance in a consistent manner over time. In accordance with this, the matrix drum must include design provisions that ensure stable, non-variable, and effectively inert matrices.
5. The surrogate waste matrix materials shall not necessitate periodic maintenance and/or inspections to validate their presence and configuration.
6. The matrix materials must be compatible with health and safety considerations such that they are not hazardous in nature, necessitating additional handling and storage precautions.
7. The selected matrix materials must be available, workable to support fabrication activities, and of reasonable cost.
8. The drum used to fabricate the surrogate matrix drum is to be of the same design and construction as used for actual packaging of DOE wastes. The drum type typically used for waste packaging purposes is a Department of Transportation (DOT) specification 17C (UN identification code UN1A2/X) 55-gallon packaging drum with a nominal 90-mil polyethylene drum liner.
9. The overall outside dimensions of the surrogate matrix drum shall be compatible with the waste NDA assay system chambers and transfer mechanisms.
10. The surrogate matrix drum must be noticeably different in appearance from actual waste drums used at the various waste characterization facilities, and from drums used for other purposes such as real-time radiography (RTR). The surrogate drum must be readily identifiable via a unique color and have a durable exterior alphanumeric identification and internal matrix code.
11. The matrix drum must be sufficiently robust in physical integrity to accommodate mechanical stresses encountered during handling and transportation. Integrity of the internal support structure and matrix must be considered in light of an expected service period of approximately 15 years.
12. The design must incorporate a means to affix an external TID for the purpose of providing control over drum contents during a measurement series.

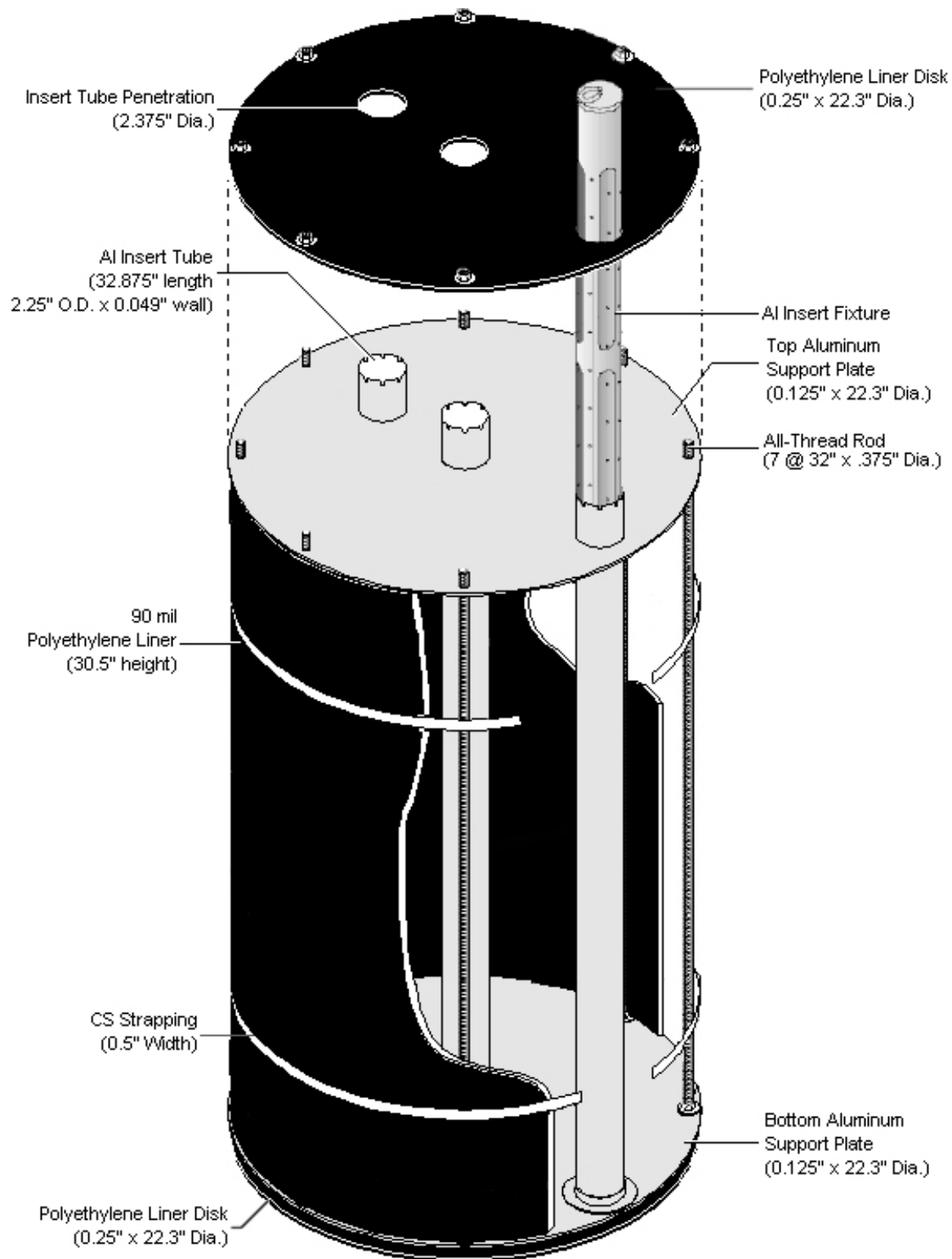


Figure C-1. NDA PDP internal support structure and noninterfering matrix drum design isometric.



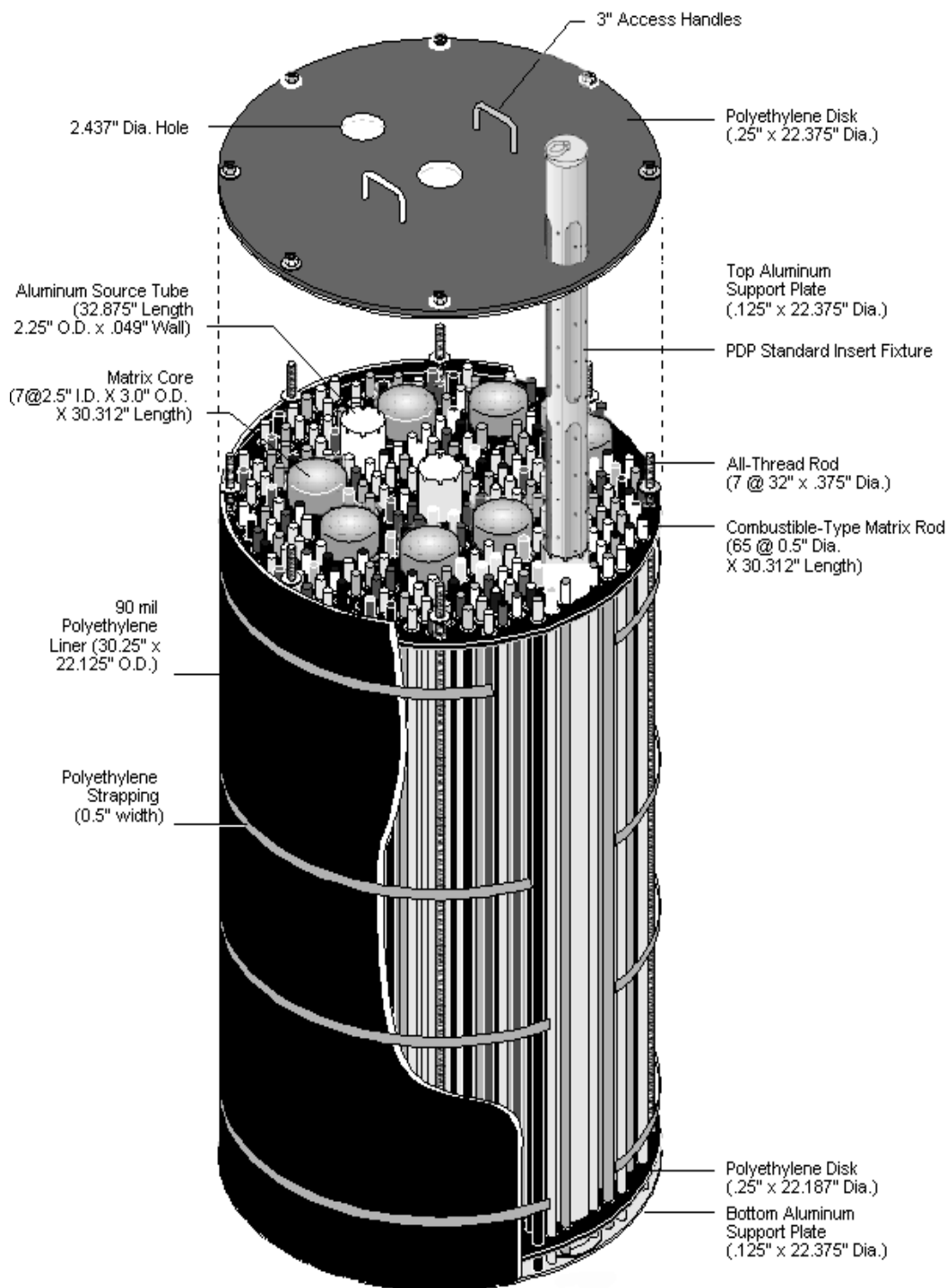


Figure C-2. NDA PDP combustibles matrix drum design isometric.

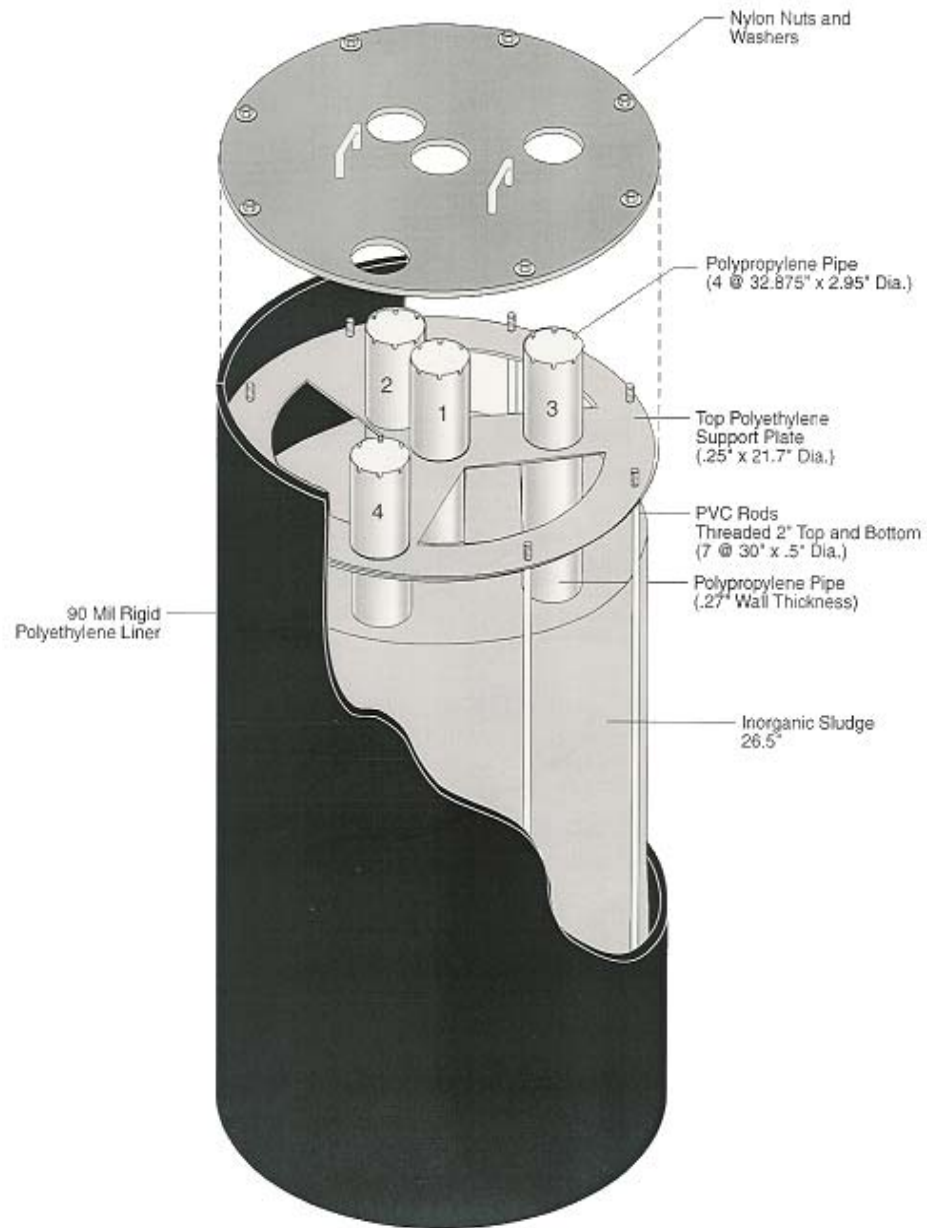


Figure C-3. NDA PDP inorganic sludge matrix drum isometric.

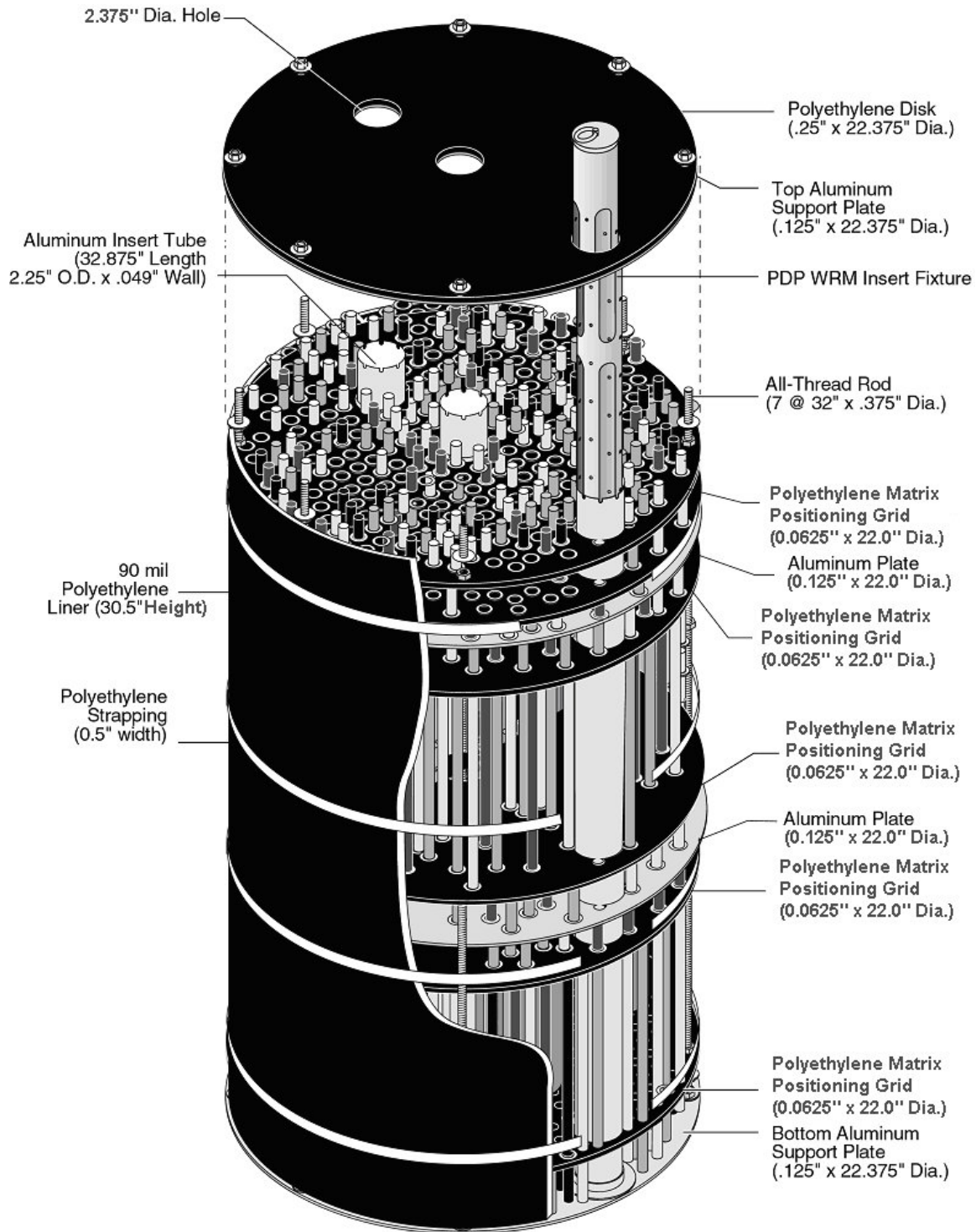


Figure C-4. NDA PDP mixed metals matrix drum isometric.

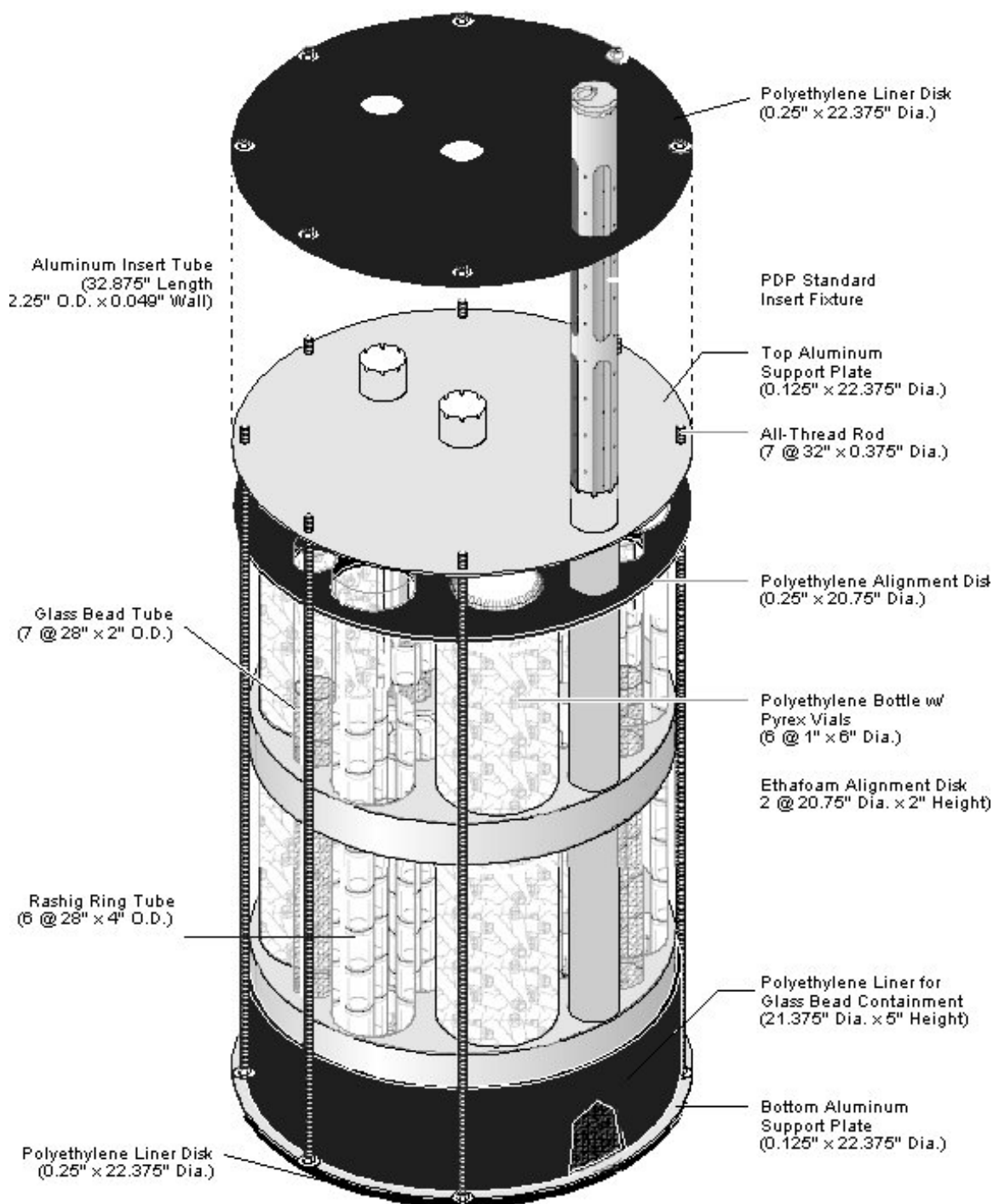


Figure C-5. NDA PDP glass matrix drum design isometric.

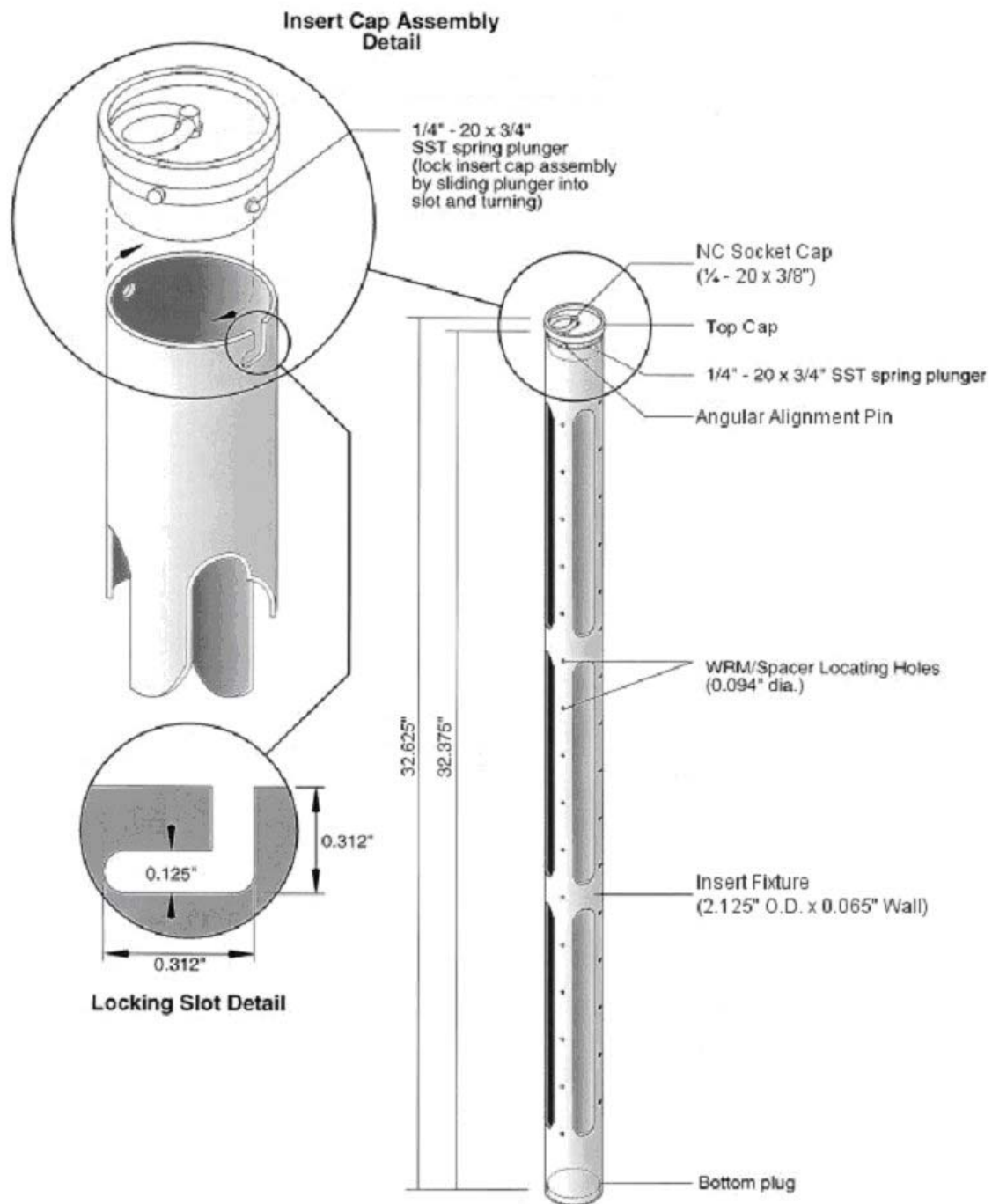


Figure C-6. NDA PDP standard insert fixture.

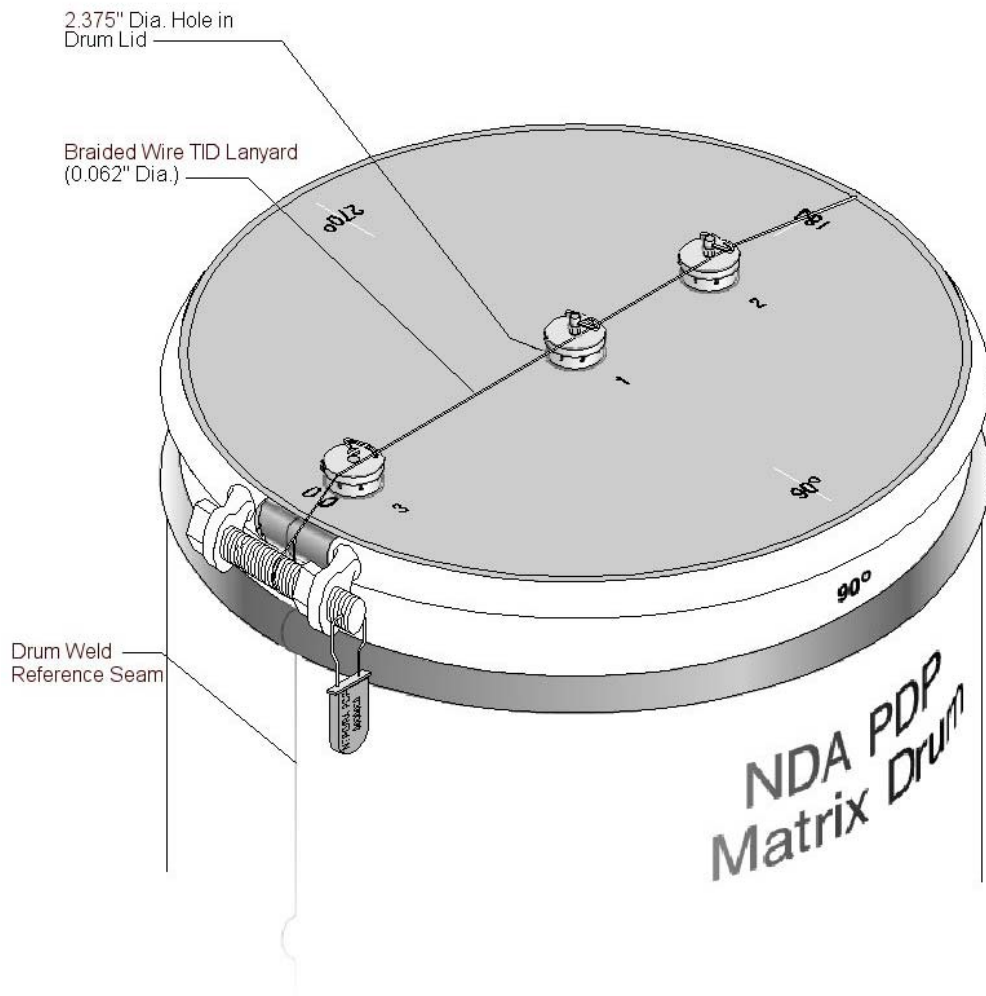


Figure C-7. NDA PDP matrix drum sample TID mechanism.

## **Appendix D**

### **Performance Demonstration Program Forms**

## Appendix D

### Nondestructive Assay PDP Sample Configuration Form

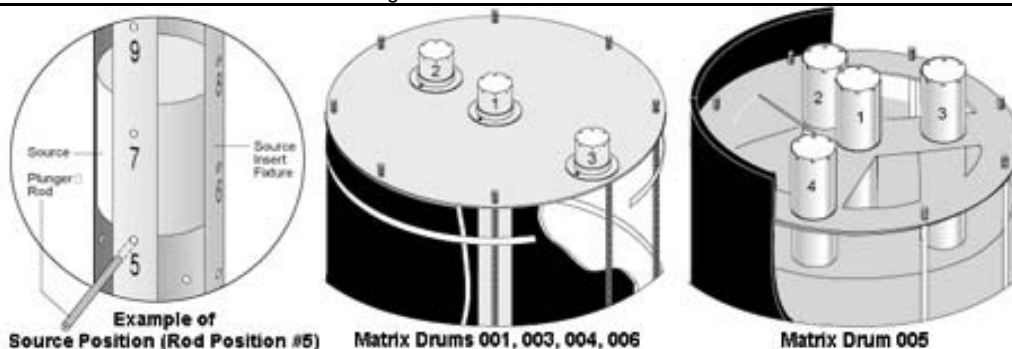
Test Facility Name:		Drum No. ___ of ___ In This Cycle.
PDP Cycle No.:	PDP Distribution (Mo/Yr):	PDP Sample ID:
Matrix Drum Serial No.:	Matrix Type:	TID No.:

Authorized: _____ Technical Liaison	_____ Date
--	---------------

PDP Standard ID	Contents of Standard			Placement		SPT Attestant Initials
	Primary Nuclear Materials	Activity (Ci) or Mass (g)	Units	Tube No. (1,2,3,4) <sup>a</sup>	Rod Position (in.) <sup>b</sup>	
Drum Total:						

Notes: a. Drums will have three or four source tubes depending on matrix simulated. (See figure below.)  
 b. Height from bottom of insert fixture in inches.

Signatures: _____ PDP Standards Custodian	_____ Date
_____ Standards Configuration Attestant	_____ Date





## Nondestructive Assay PDP Sample Custody Form

Drum Serial Number: \_\_\_\_\_ Assay Site: \_\_\_\_\_

TID Serial Number: \_\_\_\_\_

Distribution Cycle Number: \_\_\_\_\_

Comments: \_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

### Sample Preparation

Sample Preparation Date:

\_\_\_\_\_

PDP Standards Custodian

Date

Initials

Standards properly placed:

\_\_\_\_\_

Matrix drum TID properly sealed:

\_\_\_\_\_

Sample information form attached and sealed:

\_\_\_\_\_

\_\_\_\_\_

Standards Configuration Attestant

Date

Relinquished by:	Date/Time	Received by:	Date/Time

Final Disposition by:

Date/Time

Disposition

--	--	--

White: NDA PDP coordinator copy on final disposition  
Canary: SPT copy on final disposition  
Pink: NDA PDP coordinator copy  
Gold: SPT copy

## Nondestructive Assay Performance Demonstration Program Report Form

Laboratory ID: \_\_\_\_\_ Assay Facility: \_\_\_\_\_  
PDP Cycle: \_\_\_\_\_ Supplemental Cycle: \_\_\_\_\_ Replicate: \_\_\_\_\_ of \_\_\_\_\_  
Drum Serial No.: \_\_\_\_\_ Laboratory Sample ID: \_\_\_\_\_

### Final Result Summary

Parameter	Final Result	Total Uncertainty (One Standard Deviation)
Total <sup>239</sup> Pu fissile gram equivalent (g)		
Total TRU alpha activity (curies)		
Thermal Power (W)		

### Method Summary

	Identification	Classification	Associated SOP Identification (Including Revision No.)	Count Time (min)	Analysis	
					Date	Time
Method 1						
Method 2						
Method 3						

### Individual Isotope Data

Isotope	Activity Result	Uncertainty (1sd)		Quantification Method				Method Number (From Summary)
		Count	Total	Direct	Ratio	Scaling Isotope	Ratio Value	
<sup>238</sup> Pu				□	□			
<sup>239</sup> Pu				□	□			
<sup>240</sup> Pu				□	□			
<sup>241</sup> Am				□	□			
				□	□			
				□	□			
				□	□			
				□	□			

Comments:

---



---



---

Approval: \_\_\_\_\_  
Signature Title Date

## **Appendix E**

### **Statistical Basis of NDA PDP Scoring Criteria**

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### Statistical Basis of NDA PDP Scoring Criteria

#### E.1. DEFINITIONS

##### Limits and Bounds

This discussion describes two types of bounds or limits: (1) those specified as QAOs that define the *acceptable* precision limits for an NDA measurement system, and (2) the allowable bias range defined by the bounds of the 95% confidence interval. While the terms “limits” and “bounds” can be used interchangeably, to avoid confusion the term “limits” is used here only in reference to the Performance Demonstration Program (PDP) precision criteria. Similarly, the term “bounds” is used only to describe the endpoints or bounding values of calculated 95% confidence intervals for the percent recovery.

##### Point Estimate

A point estimate is the best single numerical value that is a good indicator of the underlying parameter of interest. Point estimates contrast with confidence bound estimates, which are interval estimates (since they delineate bounds on confidence intervals). For bias, the point estimate is the mean calculated percent recovery relative to the known value. For precision, the point estimate is the percent standard deviation relative to the known value.

#### E.2. PERFORMANCE CRITERIA

For a noninterfering matrix, the NDA PDP QAOs (Table E-1, column 2) specify acceptable limits for the measured precision of a nondestructive assay (NDA) system based on 15 replicate determinations. Precision, as used in the WAC and NDA PDP, is defined as the percent %RSD. The NDA PDP QAOs are related to WAC (DOE 2004) precision criteria by the maximum allowable %RSD for a noninterfering matrix of 20.0% using 15 replicates, WAC Table A-3.2. This same 20 %RSD precision limit is the same as that of the NDA PDP QAO Table E-1 column 2 at the low activity range.

The measured precision, based on 15 replicates, is only an approximation of the true system precision. Hence, implicit in each QAO limit for the measured precision is a corresponding 95% upper confidence endpoint value on the true system precision. These upper limits are stated explicitly in Table E-1, column 3. Precision criteria for NDA PDP tests for 6 replicate measurements, derived in relation to the upper confidence limits in column 3, are given in columns 4 and 5, noninterfering and interfering type matrices, respectively.

The percent recovery criteria for noninterfering matrix bias as specified in the WAC, +/- 30%, have been adopted for use in the NDA PDP, column 6. The PDP criteria for bias for the interfering type PDP sample matrices, shown in column 7 are based on the noninterfering value but are less restrictive.

Table E-1. NDA PDP performance criteria.

Activity Range in $\alpha$ -Curies	QAO for Precision (@ 15 Replicates)	Maximum Allowable Precision (95% CB of QAO)	Maximum Measured PDP Precision (@ Six Replicates)		Maximum PDP QAOs for Bias (Values for %R <sub>L</sub> and %R <sub>U</sub> for use in Equation 11) (@ Six Replicates)	
			Noninterfering	Interfering	Noninterfering	Interfering
>0 to 0.02	0.20	0.292	0.14	0.16	Low: 70 High: 130	Low: 40 High: 160
>0.02 to 0.2	0.15	0.219	0.105	0.12	Low: 70 High: 130	Low: 40 High: 160
>0.2 to 2.0	0.10	0.146	0.07	0.12	Low: 70 High: 130	Low: 40 High: 160
>2.0	0.05	0.073	0.035	0.06	Low: 70 High: 130	Low: 40 High: 160

### Precision Criteria for Noninterfering Waste Matrices

The true precision and bias of a measurement system are unknown. Estimates of the values of these parameters are acquired through random sampling of the fixed population. In the case of the NDA PDP, the fixed population is the PDP sample and the random sample is the replicate measurement set of six NDA measurements. Precision and bias are estimated using replicate assay measurement results of the total TRU  $\alpha$ -activity parameter. The more measurements acquired, the better are the precision and bias estimates of the NDA system relative to a given PDP sample configuration.

The PDP limits for measured precision of a noninterfering type matrix, determined from six replicate samples, are given in Table E-1, column 4. These values were derived from the upper confidence bounds listed in column 3, but with six replicate measurements of the PDP sample. This results in a downward adjustment of the acceptable measured precision values compared to that allowable for 15 replicates, as shown in column 2.

For example, when six replicates are used, a measured value of 18% for the relative standard deviation of an assay system in the low activity range, even though less than the 20% allowable using 15 replicates, does not mean the implicit QAO of an upper confidence bound of 29.2 has been met. In fact, the 95% one-sided upper confidence bound for this six-replicate example is approximately 38% — considerably higher than the allowable limit. Hence, the allowable measured precision with only six replicates is lower than that for 15 replicates for each  $\alpha$ -activity range.

Since the 95% confidence limit for relative standard deviation depends only on the standard deviation itself, it is possible, with a pre-specified fixed sample size, to determine ahead of time exactly how large the calculated PDP precision-point estimate value can be and still have an associated upper one-sided 95% confidence limit that meets the criteria in column 3 of Table E-1. The fourth and fifth columns of Table E-1 tabulate these maximum measured relative precision point estimate values using six replicates. The limits in columns 5 and 6 are used to compare the calculated NDA measurement system point estimate for relative standard deviation from six replicate measurements on noninterfering and interfering PDP sample matrices, respectively. (Exactly how the values for interfering matrices in columns 5 and 6 were obtained is described below.) Note that comparing the noninterfering NDA measurement system point estimate to the value in column 4 is equivalent to comparing the associated upper one-sided 95% confidence limit to the value in column 3. That is, a PDP point estimate of the value indicated in column 4 using six replicates will have a 95% upper one-sided confidence limit equal to the value in column 3.

## Calculating Limits for Measured Relative Precision

The limits specified in column 4 for the noninterfering PDP sample relative precision (standard deviation divided by the known value) are derived from confidence interval calculations for the variance (i.e., the square of the standard deviation) of a distribution. The derivation is described below.

### General Derivation

Let  $\sigma^2$  equal the true variance and let  $1 - \alpha$  equal the desired confidence value. Furthermore, let  $s^2$  equal the sample standard deviation, and  $\chi^2_{\alpha, n-1}$  equal the critical value of a chi-square distribution with  $n-1$  degrees of freedom above which  $\alpha\%$  of the distribution lies. That is, the critical value for the upper  $\alpha\%$  tail of the distribution. Then, assuming a normal distribution, a two-sided  $(1 - \alpha)\%$  confidence interval for the true variance is (e.g., Anderson 1987).

$$\frac{(n-1)s^2}{\chi^2_{\alpha/2, n-1}} < \sigma^2 < \frac{(n-1)s^2}{\chi^2_{1-\alpha/2, n-1}}. \quad (E-1)$$

Based on this formula for the two-sided interval, the upper one-sided  $(1 - \alpha)\%$  confidence limit is

$$\sigma^2 < \frac{(n-1)s^2}{\chi^2_{1-\alpha, n-1}}, \quad (E-2)$$

and the corresponding upper limit for the true percent relative standard deviation is calculated as

$$\frac{\sigma}{\mu} 100\% < \sqrt{\frac{(n-1) \frac{s^2}{\mu^2}}{\chi^2_{1-\alpha, n-1}}} 100\%, \quad (E-3)$$

where  $\mu$  is the reference (or true) mean of the distribution.

For the NDA PDP tests,  $n = 6$  and  $\chi^2_{1-\alpha, n-1} = \chi^2_{.95, 5} = 1.145$  in Equation E-3. Substituting these values and the known value of the PDP sample for  $\mu$  in this formula gives an approximate upper one-sided 95% confidence limit for the percent relative standard deviation for six replicates. If desired, this upper confidence limit can be directly compared to the numbers in column 3 of Table E-1 to determine if an assay system has met the relative precision criteria.

The numbers in column 4 of Table E-1 are derived by comparing the right portion of Equation E-3 to the appropriate number in column 3 of Table E-1 and solving for  $s/\mu$ . As an example, for the low activity range this calculation begins with the QAO required inequality

$$\sqrt{\frac{(n-1) \frac{s^2}{\mu^2}}{\chi^2_{1-\alpha, n-1}}} 100\% < 29.2\% \quad (E-4)$$

Solving for  $s/\mu$  gives

$$\frac{s}{\mu} 100\% < \sqrt{\frac{(0.292)^2 \chi_{1-\alpha, n-1}^2}{n-1}} 100\% \quad (\text{E-5})$$

which, for six samples and 95% confidence as specified in the PDP, gives

$$\frac{s}{\mu} 100\% < \sqrt{\frac{(0.292)^2 (1.145)}{5}} 100\% = 14\% . \quad (\text{E-6})$$

Again, substituting the reference (or true) value of the PDP sample for  $\mu$  indicates that a calculated relative standard deviation of 14% or less meets the QAO for relative precision in the low activity range. Since the chi-square value and  $n$  are the same for all activity levels, the column 4 values for the other activity levels are obtained simply by substituting the appropriate value from column 3 in place of 0.292 in Equation E-6.

### Precision Criteria for Interfering Waste Matrices

The WAC QAOs are specified for a “noninterfering matrix,” in other words, a waste matrix that does not have attributes that manifest themselves in the NDA measurement system as significant complicating error elements. To determine rational precision scoring criteria for the interfering type waste form, it was necessary to establish some relationship to program objectives that can be used as a basis for the NDA PDP criteria for the interfering matrix drums. There are certain program-defined limits for which assay systems are used to ensure compliance. In particular, there are the 200 fissile gram equivalent (FGE) material limits for 55-gallon containers and the TRU waste  $\alpha$ -activity definition used to discriminate TRU waste from low-level waste (LLW). At the high end, the precision of the assay system should be reasonable for waste containers approaching the 200 FGE limit to ensure that an excessive number of drums do not exceed the limit at the 95% confidence level. Similarly, the waste assay system should be sufficiently precise for containers of low TRU mass loading (i.e., in the vicinity of the 100 nCi/gram  $\alpha$ -activity criterion) to ensure that an unacceptable number of containers of TRU waste are not classified as LLW.

As a convenient base for determining precision criteria for interfering type waste matrix drums, the noninterfering compliance points in Table E-1 are used. For the low activity range the nominal compliance point for meeting the WAC precision and bias criteria is 100 mg of weapons-grade plutonium (WG Pu). An acceptable assay system should be capable of detecting and quantifying TRU waste in 55-gallon waste containers at a level of 35 mg WG Pu, ~75 nCi/g waste at 100 pounds of waste. When assaying a container at the compliance point of 100 mg WG Pu, we would like to be sure at the 95% confidence level that the assay system will not return a value less than 35 mg WG Pu. This provides reasonable protection against classifying TRU waste as LLW. Based on this rationale, two standard deviations would correspond to 65 mg (100 mg–35 mg). One relative standard deviation would therefore be 32.5mg/100 mg or 0.325. By substituting 0.325 in place of 0.292 in Equation E-6, we obtain a value of 0.155 (rounded up to 0.16) for the measured precision criterion for six replicate determinations of an interfering matrix drum in the low activity range.

Using similar reasoning, a precision criterion can be assigned to the high-mass region. In this case, the nominal compliance point used is 160 g WG Pu. When assaying a container at the compliance point of 160 g WG Pu, we would like to be sure at the 95% confidence level that the assay system will not return a

value greater than 200 g WG Pu. This provides reasonable protection against mistakenly classifying a TRU waste drum as not shippable when in fact it does not exceed the limit. Based on this rationale, two standard deviations would correspond to 40 g (200 g–160 g). One relative standard deviation would therefore be 20 g/160 g or 0.125. By substituting 0.125 in place of 0.292 in Equation E-6, we obtain a value of 0.0598 (rounded up to 0.06) for the measured precision criteria for six replicate determinations of an interfering matrix drum in the high activity range.

No compelling programmatic objectives argue for specific precision limits for the low-middle and high-middle ranges, although some thermal limits will fall into these ranges for some waste forms. Therefore, it was felt that arbitrary limits based on consistency and continuity in the use of the assay systems would be adequate for these ranges. The precision criteria for the low-middle and high-middle ranges were set at 0.12 for the RSD of six replicate determinations.

### E.3 CALCULATING CONFIDENCE BOUNDS FOR BIAS

The comparison of an assay system's performance to the bias requirements for the noninterfering and interfering PDP type samples requires calculation of the 95% two-sided confidence bounds for the true value, using the replicate measurement data set. Based on a  $t$ -distribution, the  $(1 - \alpha) \%$  two-sided confidence bounds for the true assay system mean are (assuming a normal distribution):

$$\bar{x} - t_{1-\alpha/2, n-1} \frac{s}{\sqrt{n}} < \mu < \bar{x} + t_{1-\alpha/2, n-1} \frac{s}{\sqrt{n}} \quad (E-7)$$

In terms of percent recovery, the bounds are

$$\frac{\bar{x} - t_{1-\alpha/2, n-1} \frac{s}{\sqrt{n}}}{\mu_0} 100\% < \frac{\mu}{\mu_0} 100\% < \frac{\bar{x} + t_{1-\alpha/2, n-1} \frac{s}{\sqrt{n}}}{\mu_0} 100\% \quad (E-8)$$

where  $\mu_0$  is the known (or true) value. The lower and upper bounds, calculated per Equation E-8, must be greater than  $\%R_L$  and less than  $\%R_U$ , respectively, where  $\%R_L$  and  $\%R_U$  are the appropriate lower and upper range specific QAOs from Table E-1 (column 6 or 7). Equivalently, bounds for the point estimate, total TRU  $\alpha$ -activity percent recovery, can be obtained by solving the required inequalities for percent recovery. The required inequalities are:

$$\frac{\bar{x} - t_{1-\alpha/2, n-1} \frac{s}{\sqrt{n}}}{\mu_0} 100\% > \%R_L \quad \text{and} \quad \frac{\bar{x} + t_{1-\alpha/2, n-1} \frac{s}{\sqrt{n}}}{\mu_0} 100\% < \%R_U \quad (E-9)$$

which, on solving for percent recovery, gives

$$\%R_L + \frac{t_{1-\alpha/2, n-1} \frac{s}{\sqrt{n}}}{\mu_0} 100\% < \frac{\bar{x}}{\mu_0} 100\% < \%R_U - \frac{t_{1-\alpha/2, n-1} \frac{s}{\sqrt{n}}}{\mu_0} 100\% \quad (E-10)$$

With six samples,  $n = 6$ , and the corresponding  $t$  value (for 95% two-sided confidence bounds) is 2.571. So the equation simplifies to:



$$\%R_L + \frac{1.05s}{\mu_0}100\% < \frac{\bar{x}}{\mu_0}100\% < \%R_U - \frac{1.05s}{\mu_0}100\%$$

(E-11)

## E.4 REFERENCES

Anderson, R. L. 1987, *Practical Statistics for Analytical Chemists*, New York, Van Nostrand Reinhold.

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